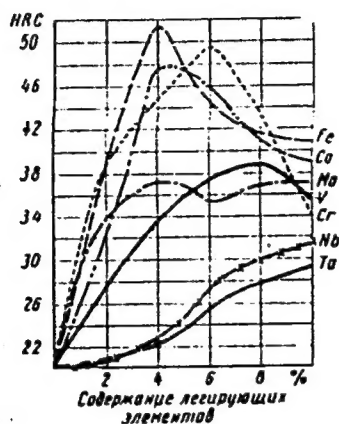


Dilatometric studies ....

S/129/63/000/002/006/014  
E193/E383

Fig. 1:



Card 4/4

L-11288-63

EWP(q)/EWT(m)/BDS--AFFTC/ASD--JD

ACCESSION NR: AP3001956

S/0226/63/000/003/0088/0098

56  
55

AUTHOR: Borok, B. A.; Golubeva, L. S.; Shchegoleva, R. P.; Ruch'yeva, N. A.

TITLE: Mechanical properties and microstructure of sintered titanium alloys

27

SOURCE: Poroshkovaya metallurgiya, no. 3, 1963, 88-98

TOPIC TAGS: sintered titanium alloys, mechanical properties, microstructure, grain size, alloying element effect, Fe, Mn, Cr, Mo, Al, V, W, Ta, Nb, Cu, Zr, Co, Ti-Al-V alloy, Ti-Al-V-Mo alloy, coreduction, oxide

ABSTRACT: Several series of binary and ternary alloys of Ti with Al, Fe, Mn, Cr, Mo, W, V, Ta, Nb, Cu, Zr, and Co were sintered from commercial-grade (99.17% pure) Ti powder and powders of 99.6% pure Fe, 99.5% pure Mn, 99.69% pure Cr, 99.54% pure Ni, 99.2% pure Co, electrolytic Cu, 99.8% pure W, 99.65% pure Mo, 99.62% pure V, 98.6% pure Nb, and 98.6% pure Ta. The Ti-Al alloys and the second series of Ti-V alloys were prepared by coreduction of oxide powders with calcium hydride. Sintered specimens had a coarse, acicular microstructure, macrograins about 1 mm in diameter, and a density of 97-99% of the theoretical. The results of mechanical tests (see Figs. 1 and 2 of Enclosure) show that all the alloying elements investigated increase the tensile strength

Card

1/52

- 7. (2/55) -

L 11288-63

ACCESSION NR: AP3001956

and decrease the ductility of sintered Ti alloys. Only in Ti-V alloys produced by coreduction of oxides does ductility increase with increasing V content. These alloys generally are more ductile than commercial titanium. The strong  $\beta$ -phase stabilizers, Fe, Mn, and Cr, which promote eutectoid transformation with the formation of intermetallic compounds, produce the highest increase in tensile strength and decrease in ductility of sintered Ti alloys. The Ti-Fe, Ti-Mn, Ti-Cr, and Ti-W alloys containing 2-10% of the following element have a metastable  $\alpha + \beta$  structure with the amount of the  $\beta$ -phase increasing with higher alloying; the  $\alpha$ -phase has an acicular Widmanstätten structure. Aluminum, an  $\alpha$ -phase stabilizer, appreciably increases the strength of sintered Ti-Al alloys without an extensive decrease in ductility. The Ti-V and Ti-Mo alloys have comparatively high tensile strength and ductility. In general, V, Al, and Mo were found to be the best alloying elements for sintered binary Ti alloys. Additional investigation of sintered Ti-Al-V alloys (produced by coreduction of the oxides) showed the Ti + 3% Al + 3% V alloy to have the best combination of mechanical properties: tensile strength of 774.2 Mn/m<sup>2</sup> [meganewton per square meter, 1 Mn/m<sup>2</sup>  $\approx$  0.1 kg/mm<sup>2</sup>], elongation 15%, reduction of area 26%, and notch toughness 25.4 joule per cm<sup>2</sup> [1 joule/cm<sup>2</sup> =  $\sim$  0.1 m-kg/cm<sup>2</sup>]. An addition of 2% Mo to this alloy increases its tensile strength to 857.5 Mn/m<sup>2</sup> without lowering ductility. These two alloys are recommended for manufacturing parts by

Card 2/3 2

ACCESSION NR: AP4040471

S/0226/64/000/003/0050/0063

AUTHOR: Borok, B. A.; Shchegoleva, R. P.; Golubeva, L. S.;  
Teplenko, V. G.; Reutova, N. P.; Ruch'yeva, N. A.

TITLE: Properties and microstructure of sintered Kh18N15 stainless steel made by joint reduction method

SOURCE: Poroshkovaya metallurgiya, no. 3 (21), 1964, 50-63

TOPIC TAGS: stainless steel, sintered stainless steel, carbonyl iron, sintered steel property, steel corrosion resistance, sintered steel structure

ABSTRACT: Investigations have been made of the properties of sintered Kh18N15 chromium-nickel stainless steel made from powder produced by the joint reduction of chromium and nickel oxides mixed with iron powders (Process A) and of steel made from mechanically mixed powders of carbonyl iron, reduced chromium, and electrolytic nickel (Process B). It was found that the density of compacts A was lower than that of B, but the latter had a very low compression strength. Adequate fluidity of powders and strength of compacts

Card 1/3

ACCESSION NR: AP4040471

make powder A a very suitable material for rolling porous strips and sheets in continuous rolling mills. Compacts B sinter more easily than compacts A, but they are much more susceptible to oxidation during the sintering. Compacts A, sintered at 1350C for 10 hr; had a density of 96—97% (compared to 71—85% for compacts B), tensile strength 47.8—53.5 dan/mm<sup>2</sup>, elongation 29.2—43.4% and impact toughness (unnotched specimens) 19.8—29 kgm/cm<sup>2</sup>. Sintered Kh18N15 steel has an austenitic structure with a low content of finely dispersed carbides. In the annealed state the steel has a high corrosion resistance; its corrosion rate in boiling 65% nitric acid is 0.1 g/m<sup>2</sup> · hr compared to 0.2 g/m<sup>2</sup> · hr for conventionally made X18H15. This is explained by a low content of impurities in powder A. Orig. art. has: 8 tables and 9 figures.

ASSOCIATION: Tsentral'nyy nauchno-issledovatel'skiy institut chernoy metallurgii (Central Scientific Research Institute of Ferrous Metallurgy)

Cord 2/3

L 2847-66 EWP(e)/EWT(m)/EPF(c)/EWA(d)/EWP(t)/EWP(k)/EWP(z)/EWP(b) LJP(c)  
 MJW/JD/WB UR/2776/65/000/043/0081/0098

ACCESSION NR: AT5022891

AUTHOR: Shchegoleva, R. P.; Reutova, N. P.; Golubeva, L. S.; Poplavskaia, V. L.;  
 Kazanskaya, L. N.

TITLE: Powdered-metal stainless chrome and chrome-nickel steels

SOURCE: Moscow. Tsentral'nyy nauchno-issledovatel'skiy institut chernoy me-  
 tallurgii. Sbornik trudov, no. 43, 1965. Poroshkovaya metallurgiya (Powder metal-  
 lurgy), 81-98

TOPIC TAGS: powder metallurgy, stainless steel, chromium steel, nickel steel,  
 corrosion resistance

ABSTRACT: It is shown that the powders of stainless chrome and chrome-nickel  
 steels in the ferritic, austenitic, and martensitic-austenitic classes, prepared  
 by the method of the combined reduction of metal oxides by means of  $\text{CaH}_2$ , are  
 suitable for the industrial fabrication of porous and compact sheets and strips  
 by the direct method of powder rolling. The flowsheet of production of these  
 powders has the following sequence: raw materials -- iron powder (carbonyl and  
 other types of Fe), chromium oxide ( $\text{Cr}_2\text{O}_3$ ), nickel (electrolytic, carbonyl)

Card 1/3

L 2847-66

ACCESSION NR: AT5022891

2

powder or  $\text{NiO}$ ,  $\text{Ni}_2\text{O}_3$ , calcium hydride ( $\text{CaH}_2$ ); charge blending (2.5 hr); reduction at  $1175^\circ\text{C}$  for 6-8 hr,  $\text{Cr}_2\text{O}_3 + 3\text{CaH}_2 = 2\text{Cr} + 3\text{CaO} + 3\text{H}_2$ ; crushing of sinter; slaking with  $\text{H}_2\text{O}$  and pulverization; hydrocyclone treatment of pulp; leaching --  $\text{Ca}(\text{OH})_2 + 2\text{HCl} = \text{CaCl}_2 + 2\text{H}_2\text{O}$ ; washing to remove  $\text{CaCl}_2$ ; centrifuging; vacuum drying,  $60-70^\circ\text{C}$ . Sintered stainless steels display high physical properties, which warrants recommending them for the fabrication of the elements and devices performing in aggressive media. When pressed under a pressure of  $10 \text{ t/cm}^2$  and subjected to deformation and heat treatment, powdered-metal stainless steels are not inferior to steels produced by the smelting method as regards their physical properties and corrosion resistance. Thus, for example, corrosion tests of Kh18N15 stainless austenite steel in a 65% solution of boiling  $\text{HNO}_3$  demonstrated the high corrosion strength of this steel, not inferior to that of deformed cast steel (corrosion rate  $0.1-0.16 \text{ g/m}^2\text{-hr}$ ). Evidently these good qualities of powdered-metal stainless steels are attributable to the low content of impurities in the powders prepared by the combined oxide reduction method. Orig. art. has: 10 figures, 9 tables.

ASSOCIATION: none

Card 2/3

L 2847-66

ACCESSION NR: AT5022891

SUBMITTED: 00

ENCL: 00

SUB CODE: MM.

NO REF SOV: 007

OTHER: 007

BVK

Card 3/3



L 2679-66 EWP(e)/EWT(m)/EWA(d)/EWP(t)/EWP(k)/EWP(z)/EWP(b) IJP(c) MJW/

ACCESSION NR: AT5022892 JD/HW

UR/2776/65/000/043/0099/0108

58  
56  
8-11

AUTHOR: Solov'yeva, Z. V.; Golubeva, L. S.; Shchegoleva, R. P.; Ruch'yeva, N. A.; Kudinova, K. G.

44,55  
44,55

44,55

TITLE: Investigation of the properties and production conditions of nichrome powder

SOURCE: Moscow. Tsentral'nyy nauchno-issledovatel'skiy institut chernoy metallurgii. Sbornik trudov, no. 43, 1965. Poroshkovaya metallurgiya (Powder metallurgy), 99-108

TOPIC TAGS: nichrome alloy, powder alloy, nonmetallic inclusion, sintering, solid solution, twinning, heat resistant alloy, resistivity

ABSTRACT: In view of the deviations observed in the technological properties of the products fabricated from the powder of Kh20N80 nichrome alloy prepared by the method of the combined reduction of metal oxides with  $\text{CaH}_2$  developed by the Central Scientific Research Institute of Ferrous Metallurgy, the authors performed a thorough investigation of the parameters of the process. Gas analyses and metallographic examinations established that nichrome powders obtained at

Card 1/3

L 2679-66

ACCESSION NR: AT5022892

oxide-reduction temperatures of 900-1100°C (for 6 hr) contain a considerable amount of non-metallic inclusions, associated with the higher content of oxygen. This condition is corrected (the oxygen content is reduced to the required minimum of 0.4% and the microstructure becomes homogeneous) by raising to 1175°C the reduction temperature and performing reduction for 6-8 hr (6 hr for 219-mm diameter retort and 8 hr for 273-mm diameter retort). However, while the powder prepared at 1175°C for 6-8 hr displays the optimal compactibility, its sinterability is much lower than in powders prepared at lower reduction temperatures (900-1100°C), which evidently is attributable to the activating effect of oxygen as well as to granulometric composition. Since, the oxygen content may not exceed 0.04%, it appears that sinterability can be improved only by altering the granulometric composition of the powder. This composition can be regulated within broad limits by pulverizing the sinter (pulp) for 0.5, 1.0, 1.5, and 2 hr. To evaluate its quality, the powdered-metal nichrome prepared on the basis of the above improvements was subjected to heat treatment and cold working and tested for physical properties. Specimens compacted under a pressure of 6.0-6.8 tons/cm<sup>2</sup> and sintered at the maximum temperature (1375°C) were found to display the highest ultimate strength and plasticity. Wire of 0.5-2.0 mm diameter fabricated from sintered briquets displays, following its heat treatment (water quenching from

Card 2/3

L 2679-66

ACCESSION NR: AT5022892

870°C), physical properties as high as those of standard nichrome wire. Following its sintering, as well as following its forging in the temperature range 1000-1200°C, the powdered-metal nichrome has the monophasic structure of a nickel-base solid solution with grain boundaries clearly revealed by etching. Following its annealing at 800 or 900°C the nichrome displays the typical structure of nickel austenite; the grain orientation changes and a large number of twins appears. In addition to their high heat resistance and resistance to oxidation at high temperatures, the products fabricated from such nichrome powder display a high resistivity (1.07-1.12 ohm-mm<sup>2</sup>/m). Orig. art. has: 10 figures, 6 tables.

ASSOCIATION: none

SUBMITTED: 00

ENCL: 00

SUB CODE: MM, IE

NO REF SOV: 007

OTHER: 004

Card

L 2680-66 ENT(m)/EWA(d)/EWP(t)/EWP(z)/EWP(b) MJW/JD

ACCESSION NR: AT5022893

UR/2776/65/000/043/0109/0114

44.58  
44.58  
44.58

AUTHOR: Shchegoleva, R. P.; Golubeva, L. S.; Ruch'yeva, N. A.; Poplavskaya, V. L.

TITLE: Investigation of the microstructure of alloy powders obtained by the combined reduction method, 6

SOURCE: Moscow. Tsentral'nyy nauchno-issledovatel'skiy institut chernoy metallurgii. Sbornik trudov, no. 43, 1965. Poroshkovaya metallurgiya (Powder metallurgy), 109-114

TOPIC TAGS: metal powder, alloy powder, nichrome alloy, grain structure

ABSTRACT: The process of the combined reduction of metal powders and oxides by means of  $\text{CaH}_2$  usually takes place in the solid phase, and then the granulometric composition of the resulting powder is chiefly determined by two factors: 1) physical properties of raw materials; and 2) the process of agglomeration of the metal particles (this process takes place in the solid phase for both fine- and coarse-grained materials). Studies of a large number of industrial batches of the powder of Kh20N80 nichrome established that distinct structural inhomogeneities appear only when the technological regimes of production are disregarded. Such

Card 1/3

L 2680-66

ACCESSION NR: AT5022893

5

inhomogeneities are manifested in the form of the presence of a second phase although no such phase was revealed by radiographic examination. A microscopic examination of the Sulin and Tula iron powders revealed, along with particles having ferrite structure, isolated particles with ferrite + pearlite structure conditioned by a higher content of C. Such nonuniformity of individual particles as regards C content also persists in Kh18N15 steel. Particles with two-phase structure have been observed in individual industrial batches of Fe-Al master alloy powder which indicates violations of the technological regime of charge blending, mixing, and reduction. The microstructural inhomogeneity of the powder of Kh18N15 steel, conditioned by its content of alloy elements, is greater if the comparatively coarse-grained Sulin and Tula iron powders are used as part of the raw materials. In this case an appreciable amount of  $\alpha$ -phase is observed in the microstructure of the large particles. If, on the other hand, this steel, as well as Kh20N80 nichrome alloy, is prepared from fine-grained raw materials, the resulting powders will display some inhomogeneity with respect to the content of alloy elements, owing to their extremely weak ferromagnetic properties. All this, however, is no reason for rejecting the powders as defective, since, being chiefly destined for processing into metallurgical products, they are subjected to sintering, which involves complete homogenization of their composition.

2/3

Card

L 2680-66

ACCESSION NR: AT5022893

Orig. art. has: 5 figures.

ASSOCIATION: none

SUBMITTED: 00

ENCL: 00

SUB CODE: MM, IE

NO REF SOV: 007

OTHER: 000

*RC*  
Card 3/3



SHCHEGOLEVA, T. A.

7

Organoboron compounds. IV. Synthesis of esters of dialkylboronic acids with the use of lithium reagents. B. M. Mishailov and T. A. Shchegoleva (N. D. Zelinski Inst. Org. Chem., Acad. Sci. USSR, Moscow). *Izv. Akad. Nauk S.S.S.R., Otdel. Khim. Nauk* 1955, 1124-5; cf. preceding abstr. — A soln. of BuLi, prepd. from 43 g. BuBr, 4.17 g. Li, and 225 ml. Et<sub>2</sub>O at 0°, treated over 1 hr. at -70° with 53 g. BuB(OBu)<sub>2</sub> in 60 ml. Et<sub>2</sub>O, stirred 4 hrs., kept overnight at this temp.; aqd. treated with dry HCl, freed of Et<sub>2</sub>O, filtered, and the filtrate freed of solvents, refiltered, and distd. yielded 74.5% Bu<sub>2</sub>BOBu, b<sub>m</sub> 122-4°, d<sub>4</sub> 0.8077. Similarly PrLi gave 67% PrBuBOBu, b<sub>m</sub> 84-6°, d<sub>4</sub> 0.7888. G. M. Kosolapoff

Chem 2 RM



SHCHEGOLEVA, T. A.

✓ Reaction of vinyl ethers with amines. A. N. Kost, A. M. Yurkevich, L. G. Yudin, and T. A. Shchegoleva (Moscow State Univ.). *Zhur. Obshchei Khim.* 25, 843-7 (1955); *J. Gen. Chem. U.S.S.R.* 25, 907-10 (1955) (Engl. translation); cf. *C.A.* 48, 13622e.—Syntheses of 2-methylquinolines from vinyl ethers and amines are described. To 9.3 g.  $\text{PhNH}_2$  and 0.2 g.  $\text{PhNH}_2\text{HCl}$  was added dropwise at 95-100° 5 g.  $\text{BuOCH}:\text{CH}_2$  and the mixt. heated gradually to 130° over 4 hrs. Distn. gave 2.7 g. quinaldine, b. 84-90° (picrate, m. 190-1°), contg. traces of  $\text{PhNH}_2$ . The same yield resulted after 25 days in sealed tube at room temp. When a trace of  $\text{Bz}_2\text{O}_2$  was used instead of  $\text{PhNH}_2\text{HCl}$ , the yield was 34%;  $\text{BF}_3\text{Et}_2\text{O}$  catalyst gave 30% yield. When 18.3 g.  $\text{PhNH}_2$ , 10 g.  $\text{BuOCH}:\text{CH}_2$ , 0.5 g.  $\text{PhNH}_2\text{HCl}$ , and 40 ml. xylene were heated 25 hrs. on a steam bath, then acidified, steam distd. to remove the solvent and  $\text{BuOH}$ , and the residue made alk. and steam distd. again, there was obtained 48% quinaldine; if the purification is made with  $\text{Ac}_2\text{O}$ , the yield rises to 57%; purification with  $\text{ZnCl}_2$  gives 33% yield. Dioxane instead of xylene gave the same yield, but without the solvent the yield reaches only 33%.  $\text{PhNH}_2$  and  $\text{EtOCH}:\text{CH}_2$

gave 30% quinaldine;  $\text{PhNH}_2$  and  $\text{iso-BuOCH}:\text{CH}_2$  gave 31% quinaldine. Heating 21.4 g.  $p\text{-MeC}_6\text{H}_4\text{NH}_2$ , 10 g.  $\text{BuOCH}:\text{CH}_2$ , and 0.5 g.  $p\text{-MeC}_6\text{H}_4\text{NH}_2\text{HCl}$  in sealed tube 20 hrs. at 100° gave 74% crude 2,6-dimethylquinoline, or 51% pure product, b. 106-7°, m. 59.5°; purification with  $\text{Ac}_2\text{O}$  instead of steam distn. gave 54% yield. Dioxane tends to raise the yield; the use of  $\text{EtOCH}:\text{CH}_2$  gave a 36% yield. Heating 21.4 g.  $o\text{-MeC}_6\text{H}_4\text{NH}_2$ , 10 g.  $\text{BuOCH}:\text{CH}_2$ , and 0.5 g.  $o\text{-MeC}_6\text{H}_4\text{NH}_2\text{HCl}$  in 50 ml. dioxane 25 hrs. at reflux gave 66% 2,8-dimethylquinoline; purification with  $\text{PhSO}_2\text{Cl}$  gave 44% pure product, while the use of  $\text{Ac}_2\text{O}$  gave 58%; pure product, b. 103-4° (picrate, m. 180-1°). To 12.3 g.  $p\text{-MeOC}_6\text{H}_4\text{NH}_2$ , 0.5 g. of its  $\text{HCl}$  salt, and 50 ml. dioxane was added slowly at reflux over 2 hrs. 10 g.  $\text{BuOCH}:\text{CH}_2$  and the mixt. refluxed 16-18 hrs. to give 15% 2-methyl-6-methoxyquinoline, b. 120-3°, m. 67°; at 80° the yield dropped to 7%; without dioxane no reaction took place. Similarly 2- $\text{C}_6\text{H}_5\text{NH}_2$  yielded after 25 hrs. at 100° 29% 2-methyl-5,6-benzoquinoline, m. 81-2° (picrate, m. 221°), if an equimolar proportion of reactants is used; a 2:1 molar ratio gave 7% yield. Refluxing 10.9 g.  $o\text{-H}_2\text{NC}_6\text{H}_4\text{OH}$ , 20 g.  $\text{BuOCH}:\text{CH}_2$ , 50 ml. dioxane, and 0.5 g.  $\text{PhNH}_2\text{HCl}$  20 hrs. gave 10% 8-hydroxyquinaldine, m. 70° (crude), m. 74° (after sublimation). Mixing 9.3 g.  $\text{PhNH}_2$ , 10 g.  $\text{BuOCH}:\text{CH}_2$ , 8.8 g. pyruvic acid, 40 ml. abs.  $\text{EtOH}$ , and 0.5 g.  $\text{PhNH}_2\text{HCl}$  resulted in vigorous reaction, which required cooling; after refluxing 10 hrs. the mixture yielded 20% 2-methylcinchoninic acid, m. 240-1° (picrate, m. 189-90°) [cf. Doebner, *Ber.* 20, 277 (1887)]. G. M. Kosolapoff

28

(3)

SHCHEGOLEVA, T. A.

7  
3  
5  
Reduction with formic acid and its derivatives. III.  
Synthesis of substituted  $\alpha$ -piperidones. A. N. Kost, T. A.  
Shchegoleva, and L. G. Yudin. *J. Gen. Chem. U.S.S.R.* 45:  
2301-8 (1955) (English translation).—See *C.A.* 50, 8410s.  
B. M. R.

RM  
MT

SHCHEGOLEVA

T. A.

Chem

6

3

1/2

Reduction with formic acid and its derivatives. III.  
 Synthesis of substituted  $\alpha$ -piperidones. A. N. Kost,  
 T. A. Shchegoleva, and L. G. Yudin (State Univ., Moscow).  
 Zhur. Obshchei Khim. 25, 2484-9 (1956); cf. C.A. 50, 8844k.  
 —Reactions of 53 g.  $\text{CH}_2=\text{CHCN}$  and 280 g.  $\text{AcCH}_2\text{CO}_2\text{Et}$   
 with  $\text{EtONa}$  in  $\text{EtOH}$  in 48 hrs. gave 50%  $\gamma$ -carbethoxy- $\beta$ -  
 oxocapronitrile, b<sub>p</sub> 143-4°, n<sub>D</sub><sup>20</sup> 1.4462, which refluxed with aq.  
 $\text{K}_2\text{CO}_3$  3.6 hrs. gave 70%  $\delta$ -oxocapronitrile, b<sub>p</sub> 108-9°, n<sub>D</sub><sup>20</sup>  
 1.4403. This (10 g.) and 34 g.  $\text{HCO}_2\text{Na}$  refluxed with 23 g.  
 100%  $\text{HCO}_2\text{H}$ , cooled, and dild., then treated with excess  
 40%  $\text{NaOH}$  gave 68% 2-methyl-6-piperidone, m. 85°, which  
 refluxed 5 hrs. with concd.  $\text{HCl}$  gave  $\delta$ -aminocaproic acid-  
 $\text{HCl}$ , m. 146-7°. A 24-hr. reaction of 20.5 g.  $\text{CH}_2=\text{CHCN}$   
 with 178 g.  $\text{MeEtCO}$  in the presence of  $\text{EtONa}$  in  $\text{EtOH}$  gave  
 27-30%  $\gamma$ -acetylacetonitrile, b<sub>p</sub> 110-11°, n<sub>D</sub><sup>20</sup> 1.4355, which  
 (10 g.) refluxed 30 hrs. with 30.5 g.  $\text{HCO}_2\text{Na}$  and 20.8 g.  
 $\text{HCO}_2\text{H}$  similarly gave 74% 2,3-dimethyl-6-piperidone, m.  
 88°. Dry  $\text{MePrCO}$  (109 g.) treated with 3 ml. 10%  $\text{EtONa}$ -  
 $\text{EtOH}$ , followed by 17.1 g.  $\text{CH}_2=\text{CHCN}$  added over 1.5 hrs.  
 and heating 8 hrs. on a steam bath, gave after neutraliza-  
 tion with  $\text{AcOH}$  26%  $\gamma$ -acetylacetonitrile (I), b<sub>p</sub> 115-16°, n<sub>D</sub><sup>20</sup>  
 1.4381, d<sub>4</sub><sup>20</sup> 0.9024. To 30 g.  $\text{AcCH}_2\text{CO}_2\text{Et}$  and 4 ml. 10%  
 $\text{EtONa-EtOH}$  was added at 10-12° in 15 min. 10 g.  $\text{CH}_2=\text{CHCN}$ ;  
 after 4 hrs. the soln. was acidified with  $\text{AcOH}$  and  
 dild., yielding 71%  $\gamma$ -acetyl- $\gamma$ -carbethoxycapronitrile, b<sub>p</sub>  
 164-4.5°, n<sub>D</sub><sup>20</sup> 1.4502, d<sub>4</sub><sup>20</sup> 1.0518, which refluxed 4 hrs. with  
 aq.  $\text{Na}_2\text{CO}_3$  gave 21% I, b<sub>p</sub> 142-3°. I (11 g.) refluxed 40  
 hrs. with 18.4 g.  $\text{HCO}_2\text{H}$  and 27.1 g.  $\text{HCO}_2\text{Na}$  then treated  
 with 12.2 g.  $\text{HCO}_2\text{H}$  and refluxed 23 hrs., gave after usual  
 treatment 50% 2-methyl-3-ethyl-6-piperidone, b<sub>p</sub> 152-5°.

(OVER)

6

# REDUCTION WITH FORMIC ACID

m. 40-3°. Similarly  $\gamma$ -methyl- $\gamma$ -isobutylvaleronitrile gave 60% 3,3-dimethyl-2-isopropyl-6-piperidone, m. 111-12°. Similarly 10 g. 2-(2-cyanoethyl)cyclohexanone refluxed 20 hrs. with 16.1 g.  $\text{HCO}_2\text{H}$  and 23.8 g.  $\text{HCO}_2\text{Na}$  gave 72% *trans*-octahydro- $\alpha$ -quinolone, m. 151°; no octahydro deriv. is formed with  $\text{HCO}_2\text{H}$  alone. Refluxing the product with concd.  $\text{HCl}$  5 hrs. gave  $\beta$ -(2-aminocyclohexyl)propionic acid- $\text{HCl}$ , m. 173-4°, from which was prepd. *trans*- $\beta$ -(2-benzamidocyclohexyl)propionic acid, m. 200°, by action of  $\text{BzCl}$ . To 10 g.  $\text{BzCH}_2\text{CH}_2\text{CN}$ , 200 ml. 3%  $\text{H}_2\text{O}_2$ , and 2 ml. 6*N*  $\text{NaOH}$  was added enough  $\text{EtOH}$  to form a soln., which heated 5 hrs. at 50-60°, yielded after concn. and neutralization 45%  $\text{BzCH}_2\text{CH}_2\text{CONH}_2$ , m. 125°; a 30% yield resulted from keeping the nitrile in concd.  $\text{H}_2\text{SO}_4$  2 hrs. The amide (5 g.) refluxed 40 hrs. with 10.2 g.  $\text{HCO}_2\text{Na}$  and 7 g.  $\text{HCO}_2\text{H}$  gave after cooling, removal of tar and addn. of concd.  $\text{NaOH}$ , a resinous mass which heated briefly with 1:1  $\text{HCl}$  gave 30% 2-phenyl-6-pyrrolidone, m. 108°, this refluxed 10 hrs. with concd.  $\text{HCl}$  gave  $\gamma$ -amino- $\gamma$ -phenylbutyric acid- $\text{HCl}$ , m. 179°. Benzoylpropionitrile under the above conditions gives but a very poor yield of the phenylpyrrolidone owing to formation of much tar. Heating  $\beta$ -benzoylpropionitrile with  $\text{HCONH}_2$  and a little  $\text{HCO}_2\text{NH}_2$ , 6 hrs. at 160° gave a red solid, decomp. 250°. G. M. K.

2  
2  
3  
lost

Shegoleva

Chem <sup>1</sup> Organoboron compounds. IX. Action of phosphorus pentachloride on esters of dialkylboric and alkylboric acids. Synthesis of dialkylboron chlorides and esters of alkyl-chloroboric acids. B. M. Mikhailov and T. A. Shegoleva. Bull. Acad. Sci. U.S.S.R. Div. Chem. Sci. 1956, 501-2 (Engl. translation).—See C.A. 50, 16694c B. M. R.

2

411

E-2

USSR/ Organic Chemistry - Synthetic organic chemistry

Abs Jour : Referat Zhur - Khimiya, No 4, 1957, 11792

Author : Mikhaylov B.M., Vavar V.A. (Communication 8)  
Mikhaylov B.M., Shchegolova T.A. (Communication 9)

Inst : Department of Chemical Sciences, Academy of Sciences USSR

Title : Organic Boron Compounds. Communication 8. Synthesis and Properties of Diaryl-Boric Acids. Communication 9. On Action of Phosphorus Pentachloride on Esters of Dialkyl-Boric and Alkyl-Boric Acids. Synthesis of Dialkyl Boro-chlorides and Esters of Alkyl-Chloroboric Acids.

Orig Pub : Izv. SSSR, Otd, khim. n, 1956, No4, 451-456; 508-509.

Abstract : Communication 8. Description of synthesis of  $(\alpha\text{-C}_{10}\text{H}_7)_2\text{BOH}$  (I),  $(p\text{-BrC}_6\text{H}_4)_2\text{BOH}$  (II) and  $(p\text{-ClC}_6\text{H}_4)_2\text{B(OH).H}_2\text{O}$  (III) and study of the properties of the acids and their derivatives.  $(\alpha\text{-C}_{10}\text{H}_7)_2\text{BOC}_4\text{H}_9\text{-iso}$  (IV),  $(p\text{-BrC}_6\text{H}_4)_2\text{BOC}_4\text{H}_9\text{-iso}$  (V) and  $(p\text{-ClC}_6\text{H}_4)_2\text{BOC}_4\text{H}_9\text{-iso}$  (VI) were

Card 1/6

USSR/ Organic Chemistry - Synthetic organic chemistry

E-2

Abs Jour : Referat Zhyr - Khimiya, No 4, 1957, 11792

Residue after removal of solvents is diluted with isopentane, yield of IV 63%, MP 104-105° (from n-hexane). From filtrate, esterification of remaining iso-C<sub>4</sub>H<sub>9</sub>OH, were obtained 15% ( $\alpha$ -C<sub>10</sub>H<sub>7</sub>)BOC<sub>4</sub>H<sub>9</sub>-iso, BP 166-168°/6 mm,  $d_4^{20}$  0.9777. To a suspension of 10 g IV in 20 ml CH<sub>3</sub>OH are added 15 ml of 30% NH<sub>4</sub>OH. Obtained 97.5%  $[(\alpha\text{-C}_{10}\text{H}_7)_2\text{B}(\text{OH})_2] \text{NH}_4$  (XI), MP 107-108° (from CH<sub>3</sub>OH). Suspension of 4.94 g XI in 30 ml ether acidified with 5 ml HCl (1:1). From ether layer isolated 71.7% I, MP 114.5-115° (from petroleum ether). Solution of 2 g I in 5 ml SOCl<sub>2</sub> boiled 2 hours. After removal excess SOCl<sub>2</sub> obtained 98% X, MP 190-192° (from benzene + petroleum ether). From 0.65 mole p-BrC<sub>6</sub>H<sub>4</sub>MgBr (1 M solution) and 0.25 mole VII, after stirring for 8 hours at -75°, treatment with 450 ml of 5% HCl and esterification, were obtained 39% V, BP 161-163°/1 mm, (in N<sub>2</sub> current) and 37% p-BrC<sub>6</sub>H<sub>4</sub>B(OC<sub>4</sub>H<sub>9</sub>-iso) BP 109-110°/1 mm,  $d_4^{20}$  1.1583. 2.25 g V mixed by shaking with 5 ml 30%

Card 3/6

USSR/ Organic Chemistry - Synthetic organic chemistry

E-2

Abs Jour : Referat Zhur - Khimiya, No 4, 1957, 11792

washed with water and dried over  $\text{CaCl}_2$ . Yield 1.1 g, MP  $76-78^\circ$  (reprecipitated from alcohol with water). 2.85 g VI dissolved in 8.4 ml 1.09 N NaOH. Water slowly driven off in vacuum at  $60-70^\circ$ . Residue washed with  $\text{C}_6\text{H}_6$ . Obtained 2.2 g  $((p\text{-ClC}_6\text{H}_4)_2\text{B}(\text{OH})_2)\text{Na}$  (XV). From 0.3 g XV isolated on acidification 0.25 g III.

Communication 9. Study of interaction of  $\text{PCl}_5$  with  $(n\text{-C}_4\text{H}_9)_2\text{BOC}_4\text{H}_9\text{-n}$  (XVI) and with  $(n\text{-C}_4\text{H}_9)(n\text{-C}_3\text{H}_7)\text{BOC}_4\text{H}_9\text{-n}$  (XVII). Reaction with XVI or XVII takes place with formation of  $(n\text{-C}_4\text{H}_9)_2\text{BCl}$  (XVIII) or, respectively,  $(n\text{-C}_4\text{H}_9)(n\text{-C}_3\text{H}_7)\text{BCl}$  (XIX),  $\text{C}_4\text{H}_9\text{Cl}$  and  $\text{POCl}_3$ . It was also found that  $(n\text{-C}_4\text{H}_9)_2\text{B}(\text{OC}_4\text{H}_9\text{-n})$  (XX) and  $(n\text{-C}_4\text{H}_9)(n\text{-C}_3\text{H}_7)\text{B}(\text{OC}_4\text{H}_9\text{-n})$  (XXI) react readily with one equivalent of  $\text{PCl}_5$ , forming, respectively,  $(n\text{-C}_3\text{H}_7)\text{B}(\text{OC}_4\text{H}_9\text{-n})\text{Cl}$  (XXII) and  $(n\text{-C}_4\text{H}_9)\text{B}(\text{OC}_4\text{H}_9\text{-n})\text{Cl}$  (XXIII),  $\text{C}_4\text{H}_9\text{Cl}$  and  $\text{POCl}_3$ . To 16.8 g

Card 5/6



MIKHAYLOV, B.M.; KOZMINSKAYA, T.K.; BLOKHINA, A.N.; SHCHEGOLEVA, T.A.

Boron organic compounds. Part 10. Complex nature of salts of boron-organic acids. Izv.AN SSSR Otd.khim.nauk no.6:692-695 Jo '56.  
(MIRA 9:9)

1.Institut organicheskoy khimii imeni N.D.Zelinskogo Akademii nauk SSSR.

(Boronium salts)

*Shchegoleva, T. A.*

Organoboron compounds synthesis of trialkyl compounds  
of boron from the esters of dibutylboric acid. B. M. MI-  
khailov and T. A. Shchegoleva, Proc. Acad. Sci. U.S.S.R.,  
Sect. Chem. 108, 281-3 (1968) (English translation) - See  
C.A. 51, 1028c.

5/4E4J  
2/4E3d

cm  
gmb MT

Shchegoleva, T.A.

Organoboron compounds. Synthesis of trialkyl com-  
pounds of boron from the ester of dibutyriboric acid. B. M.  
Mikhailov and T. A. Shchegoleva. Doklady Akad. Nauk  
S.S.S.R. 108, 481-2 (1964). To  $\text{Et}_2\text{O}$  soln. of BuLi from  
 2.6 g. Li and 27.4 g. BuCl in 180 ml.  $\text{Et}_2\text{O}$  was added at  
 $-70^\circ$  27.8 g.  $\text{Bu}_2\text{BOBu}$  over 40 min.; after stirring 3 hrs. with  
 the mixt. was left overnight, after which it was sepd. with  
 dry HCl, filtered of Li halides and distd.; the fraction, bp  
 $114-19^\circ$ , was shaken with 10% NaOH, the 2 layers were  
 sepd. and the org. layer was freed of BuOH and  $\text{H}_2\text{O}$  by  
 distn.; the residue was dild. with 20 ml.  $\text{H}_2\text{O}$ , and the org.  
 layer which sepd. was azeotropically dried with  $\text{CaH}_2$  and  
 distd., yielding 33%  $\text{Bu}_3\text{B}$ , bp  $108-10^\circ$ . Similarly,  $\text{PrLi}$   
 from 4.85 g. Li with 50 g.  $\text{Bu}_2\text{BOBu}$  gave 50%  $\text{Bu}_2\text{BPr}$ ,  
 bp  $78-80^\circ$ , and 3.5 g. unreacted ester. Pure  $\text{Bu}_2\text{BPr}$  b,  $77-$   
 $9^\circ$ , d<sub>4</sub> 0.7481. When this was fractionated slowly through a  
 20-plate column, there was obtained an appreciable amt. of  
 $\text{Pr}_2\text{B}$ , b,  $60.5-4^\circ$ , and  $\text{Bu}_2\text{B}$ , bp  $98-8^\circ$ . Reaction of  $\text{EtLi}$   
 from 4.45 g. Li with 44.8 g.  $\text{Bu}_2\text{OBU}$  at  $-25^\circ$  similarly gave  
 a series of fractions from which was isolated 26%  $\text{Bu}_2\text{BEt}$ ,  
 b,  $67-8^\circ$ . The analysis of the B compds. was simplified  
 (cf. Snyder, et al., C.A. 32, 1646) by the following tech-  
 nique. The sample (0.1-0.2 g.) is treated with 1 ml. 5N  
 NaOH and 5 ml. 30%  $\text{H}_2\text{O}_2$ ; after the vigorous reaction  
 subsides, there is added 2 ml. 5N NaOH and 3 ml. 30%  $\text{H}_2\text{O}_2$ ,  
 and the mixt. refluxed 20 min.; on cooling the excess HCl is  
 neutralized by N NaOH with methyl orange indicator,  
 treated with mannitol and titrated with 0.1N NaOH with  
 phenolphthalein indicator.

G. M. Kosolapoff

Distr: 4E4j/4E2c(j)/  
4E3d

Organoboron compounds XIX. Synthesis of alkyl-  
boron dichlorides. B. M. Mikhailov and T. A. Shche-  
golova (N. D. Zhenskii Inst. Org. Chem., Moscow).  
Izv. Akad. Nauk S.S.S.R., *Chem. Nauk* 1957,  
1080-5; cf. C.A. 50, 16664c; 52, 4532f. To 150 g. B(OMe)<sub>3</sub>  
in 400 ml. Et<sub>2</sub>O was added at -76° over 9 hrs. EtMgBr  
from 36 g. Mg and on the following day (all operations  
under N) the mixt. treated with dil. HCl, and the org. layer  
freed of Et<sub>2</sub>O, and slowly distd. with BuOH until the temp.  
of the distillate reached 116° yielding 53% EtB(OBu)<sub>2</sub>.  
b<sub>1</sub> 70-1°, d<sub>20</sub> 0.8224. Similarly was prepd. 50% iso-PrB-  
B(OBu)<sub>2</sub>, b<sub>1</sub> 78-8.5°, d<sub>20</sub> 0.8187. Slow distn. of EtB-  
(OBu)<sub>2</sub> and hexyl alc. gave 83.5% EtB(OC<sub>6</sub>H<sub>13</sub>)<sub>2</sub>, b<sub>1</sub> 130-1°,  
d<sub>20</sub> 0.8302; similarly was prepd. 80% PrB(OC<sub>6</sub>H<sub>13</sub>)<sub>2</sub>, b<sub>1</sub> 125-6°,  
d<sub>20</sub> 0.8283, and 72% iso-PrB(OC<sub>6</sub>H<sub>13</sub>)<sub>2</sub>, b<sub>1</sub> 49-  
130-1°, d<sub>20</sub> 0.8268. To 23.5 g. EtB(OBu)<sub>2</sub> was added over 1 hr.  
31.8 g. PCl<sub>5</sub> yielding on distn. 78% EtB(OBu)Cl, b<sub>1</sub> 49-  
53°, d<sub>20</sub> 0.9030, BuCl, and POCl<sub>3</sub>. Similar addn. to 32.2 g.  
EtB(OC<sub>6</sub>H<sub>13</sub>)<sub>2</sub> of 55 g. PCl<sub>5</sub> and refluxing 0.5 hr. gave 60%  
EtBCl, b<sub>1</sub> 49-51°, which ignites in air and is easily hy-  
drolyzed; the same formed in 40% yield from 17.5 g. Et-  
B(OBu)Cl and 24.5 g. PCl<sub>5</sub>. EtB(OC<sub>6</sub>H<sub>13</sub>)<sub>2</sub> (54 g.) with  
46.5 g. PCl<sub>5</sub> gave 3.4 g. EtBCl, 19.5 g. EtB(OC<sub>6</sub>H<sub>13</sub>)Cl,  
b<sub>1</sub> 63-5°, and 21% EtB(OC<sub>6</sub>H<sub>13</sub>)<sub>2</sub>. PrB(OC<sub>6</sub>H<sub>13</sub>)<sub>2</sub> (31.35 g.)  
with 51.8 g. PCl<sub>5</sub> gave 73.5% PrBCl, b<sub>1</sub> 73-5°, which also  
ignites in air. Similarly, 28.5 g. iso-PrB(OC<sub>6</sub>H<sub>13</sub>)<sub>2</sub> and  
46.7 g. PCl<sub>5</sub> gave 70% iso-PrBCl, b<sub>1</sub> 70.5-73°, which  
ignites in air. XX. N-Substituted phenyldiaminoboranes and  
B-phenylborazoles. B. M. Mikhailov and P. M. Aronovich  
Ibid. 1123-5; cf. C.A. 51, 4985h. To 41.4 g. EtNH<sub>2</sub> in  
C<sub>6</sub>H<sub>6</sub> was added at -30° 31.8 g. PhBCl<sub>2</sub> in 30 ml. C<sub>6</sub>H<sub>6</sub>  
and after warming to room temp. 2 hrs. the mixt. filtered  
from EtNH<sub>2</sub>.HCl and evapd. yielding 3.5 g. B,B,B-triphenyl-  
N,N,N-triethylborazole, m. 200-3°, the mother liquor from  
this gave PhB(NHEt)<sub>3</sub> (I), b<sub>1</sub> 97-8°, d<sub>20</sub> 0.924, n<sub>D</sub><sup>20</sup> 1.5120;

*B. M. MIKHAILOV + T. A. SHCHESOLEVA*

The latter is readily attacked by  $H_2O$  and atm.  $O$ . The residue after the distn. gave more triphenyltriethylborazole for a total yield of 21%; the pure substance, m. 205-7° ( $C_6H_5$ -petr. ether); the borazole is stable in air and is slowly attacked by hot alk.  $H_2O_2$ . Heating I to 210° 2 hrs. gave 17.3% triphenyltriethylborazole. Addn. of 12.7 g.  $PhBCl_2$  to 20.5 ml.  $PhNH_2$  in  $C_6H_6$  as above gave 54.7%  $PhB(NHPh)_3$ , m. 83.5-5.5° (hexane-isopentane), which is rapidly hydrolyzed by  $H_2O$ . Heating this to 270° gave  $PhNH_2$  and 31.7% hexaphenylborazole, m. above 360° ( $C_6H_6$ ).  $PhBCl_2$  and  $Et_3NH$  similarly gave 14.1%  $PhB(NEt_3)_3$ , b.p. 70-2°. All work was done under N. XXI. New methods of synthesis of borazole derivatives. B. M. Mikhailov and T. V. Kostroma. *Ibid.* 1125-7. —  $p$ - $MeC_6H_4B(OCH_3CHMe)_3$  (58 g.) with 48.8 g.  $PCl_5$  gave 87.3%  $p$ - $MeC_6H_4BCl(OCH_3CHMe)_2$ , b. 113-15°,  $n_D^{20}$  1.5017,  $d_4^{20}$  0.9919. (iso-BuO)BPhCl (17.3 g.) treated with  $NH_3$  in  $Et_2O$  gave after sepn. of  $NH_4Cl$  and evapn. 3.85 g.  $B, B, B$ -triphenylborazole, m. 180-2°, and 78%  $PhB(OBu-iso)_3$ , b. 124-5°. Pure triphenylborazole m. 181-2.5° ( $C_6H_5$ -isopentane);  $PhBCl_2$  in  $C_6H_6$  with  $NH_3$  gave 66% above triphenylborazole, m. 180-2°. Similarly were prepd. from the appropriate chloro esters 83%  $B, B, B$ -tri( $p$ -tolyl)borazole, m. 189-90°, and 84.9% tri(1-naphthyl)borazole, m. 185-7°, while the use of appropriate amines gave 72.0%  $B, B, B$ -triphenyl- $N, N, N$ -triethylborazole, m. 205-6°, and 46.5%  $B, B, B$ -tri(1-naphthyl)- $N, N, N$ -triethylborazole, m. 269-71°. G. M. Kosolapoff

8  
2 may  
3

$\frac{2}{2}$

*dm*

AUTHORS:

Mikhaylov, B. M. Shchegoleva, T. A. SOV/62-58-6-23/37

TITLE:

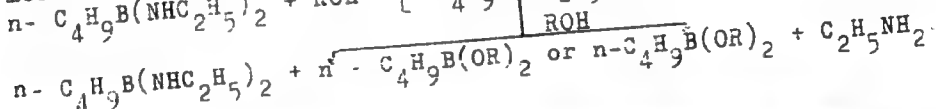
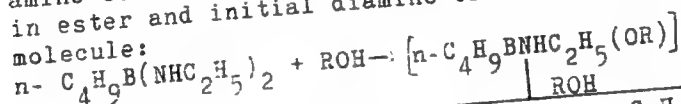
On the Effect Produced by Primary Amines Upon the Esters of  
Alkyl-chloroboric Acid (Odeystvii pervichnykh aminov na efiry  
alkilkhlorbornykh kislot)

PERIODICAL:

Izvestiya Akademii nauk SSSR, Otdeleniye khimicheskikh nauk,  
1958, Nr 6, pp. 777-779 (USSR)

ABSTRACT:

In the present report the results obtained by the investigation  
of the effect produced by primary amines (of ethylamine and  
aniline) and upon alkyl-chloroboric acid are described. Under  
the action of these primary amines the esters of alkyl-  
chloroboric acids are transformed into esters of the alkyl-  
boric acids and into N-substituted alkyl boron diamines  
(formulae I-IV). Reaction develops by way of the stage of  
amino-ester formation (IV). This ester is symmetrized either  
in ester and initial diamine or it reacts with the 2. alcohol  
molecule:



Card 1/2

On the Effect Produced by Primary Amines Upon the  
Esters of Alkyl-Chloroboric Acid

SOV/62-58-6-23/37

There is Soviet reference.

ASSOCIATION: Institut organicheskoy khimii im. N. D. Zelinskogo  
Akademii nauk SSSR (Institute of Organic Chemistry imeni  
N. D. Zelinskiy, AS USSR)

SUBMITTED: January 17, 1958

1. Alkyl chloroboric acid esters---Chemical reactions
2. Amines---Chemical reactions

Card 2/2

AUTHORS. Mikhaylov, B. M., Shchegoleva, T. A. SOV/62-58-7-11/26

TITLE. Organic Boron Compounds (Bororganicheskiye soyedineniya)  
Communication 25: On the Action of Organic Acids on the n-Butyl  
Esters of n-Chloro Propyl Boric Acid (Soobshcheniye 25. O  
deystvii organicheskikh kislot na n.butilovyy efir n.propil-  
khlorbornoj kisloty)

PERIODICAL: Izvestiya Akademii nauk SSSR, Otdeleniye khimicheskikh nauk,  
1958, Nr 7, pp 860 - 865 (USSR)

ABSTRACT. The properties of the esters of chloro aryl boric acids and  
chloro alkyl boric acids (produced by means of the interaction  
of equimolecular amounts of esters or organoboric acids and  
phosphorus pentachloride) (Refs 1,2) have hitherto been little  
investigated. In the present paper the results of the inves-  
tigation of the reactions between the n-butyl ester of chloro  
propyl boric acid and organic acids is discussed. On the action  
of acetic acid on the butyl ester of chloro propyl boric acid  
the n-butyl esters of n-butyl boric acid, n.boron propyl pyro-  
acetic anhydride, chlorobutyl and butyl acetic ester are formed.  
By the action of propionic acid on the n-butyl ester of the

Card 1/2



Organic Boron Compounds. Communication 25: On the SOV/62-58-7-11/26  
Action of Organic Acids on the n-Butyl Esters of n-Chloro Propyl Boric Acid

n-chloro propyl boric acid the n-butyl ester of n-propyl boric acid, n-boro propyl pyro propionic anhydride, chlorobutyl and butyl ester of propionic acid are formed. The authors also explained the mechanism of the reactions between organic acids and the ester of n-chloro propyl boric acid. The n-butyl ester of n-propyl boric acid reacts with acetic anhydride in the presence of hydrogen chloride with a simultaneous formation of n-boron propyl pyro acetic anhydride, butyl acetic ester, chloro butyl and chloro acetyl. There are 6 references, 4 of which are Soviet.

ASSOCIATION: Institut organicheskoy khimii im.N.D.Zelinskogo Akademii nauk  
SSSR(Institute of Organic Chemistry imeni N.D.Zelinskiy, AS USSR)

SUBMITTED: December 23, 1956

Card 2/2

*SECRET*

AUTHORS: Nikitin, A. M., Tolstunov, M. D., Aronovich, P. M., NO-1-2/20  
Shestakova, T. A., Ikhvatov, A. .,

TITLE: An Investigation of Boron Organic Compounds as Scintillators (Issledovanie stsvetlilyatorov, sodержashchikh bororganicheskiye soedineniya)

PERIODICAL: Izvestiya AN SSSR Seriya Fizicheskaya, 1958, Vol. 22, No. 1, pp. 12-15 (USSR)

ABSTRACT: The authors investigated a number of organoboron compounds (some of them were for the first time obtained in reference 3) for the purpose of determining the possibility of using them for the recording of slow neutrons. The esters of alkyl- and dialkyl boric acid were investigated on their introduction into a liquid scintillator - a p-terphenyl-solution. It became evident that the intensity of the  $\gamma$ -scintillation of the latter does almost not change. Triethylborate used in the practice of nuclear physics weakens the  $\gamma$ -luminescence of the p-terphenyl-solution by 50%. It is shown that the esters of aryl- and diarylboric acid on their introduction into a p-terphenyl-solution cause a considerable weakening of the scintillation of the latter (50-90%). The organoboron compounds with aryl-substituents themselves possess a weak  $\gamma$ -luminescence.

Card 1/2

An Investigation of Boron Organic Compounds Containing Scintil- 4.5-1-1/20  
lators.

There are 1 table, and 6 references, 3 of which are blank.

ASSOCIATION: Institute for Organic Chemistry imeni N.D. Zelinskiy AS USSR  
(Institut organicheskoy khimii im. N. D. Zelinskogo AN SSSR,  
Institute for Physics imeni P.N. Lebedev AS USSR (Fizicheskii  
institut im. P.N. Lebedeva. AN SSSR;

AVAILABLE: Library of Congress

1. Chemistry 2. Boron compounds--Application

Card 2/2

NIKITINA, A.N.; GALANIN, M.D.; ARONOVICH, P.M.; SHCHEGOLEVA, T.A.;  
MIKHAYLOV, B.M.

Analysis of scintillators containing boron organic compounds.  
Izv. AN SSSR. Ser. fiz. 22 no.1:14-20 Ja '58. (MIRA 11:2)

1. Institut organicheskoy khimii im. N.D. Zelinskogo AN SSSR i  
Fizicheskij institut im. P.N. Lebedeva AN SSSR.  
(Scintillation counters)  
(Nuclear physics--Instruments)

SECHEGILEVA, T. A., Candidate of Chem Sci (diss) -- "The synthesis and transformation of organoboron compounds of the aliphatic series". Moscow, 1959. 9 pp (Acad Sci USSR, Inst of Organic Chem im N. D. Zelinskiy), 130 copies (KL, No 21, 1959, 112)

SOV/62-59-2-30/40

5(3)

AUTHORS:

Mikhaylov, B. M., Shchegoleva, T. A.

TITLE:

Effect of Selenium on Boron Trialkyls (Deystviye selena na bortrialkily)

PERIODICAL:

Izvestiya Akademii nauk SSSR, Otdeleniye khimicheskikh nauk, 1959, Nr 2, pp 356-357 (USSR)

ABSTRACT:

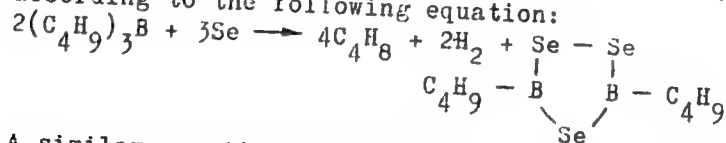
In the present news in brief the authors report on the behavior of boron trialkyls towards selenium. It was found that they react with selenium in a very peculiar way. On heating of the mixture of tri-n-butyl boron and selenium a gradual decomposition of selenium was observed at 220-250° which is accompanied by an intense separation of gaseous products. As a result of the reaction  $C_8H_{18}B_2Se_3$  is formed. On the hydrolysis of this compound n-butyl boric acid was precipitated which indicates the occurrence of a B-C bond. It is assumed that the compound obtained has a cyclic structure (Ref 1). According to the rules for the nomenclature of saturated 5-membered heterocyclic systems (Ref 3), this compound was called 3,5-di-n-butyl-3,5-diboron-1,2,4-triselenolane. The gas separated during the re-

Card 1/3

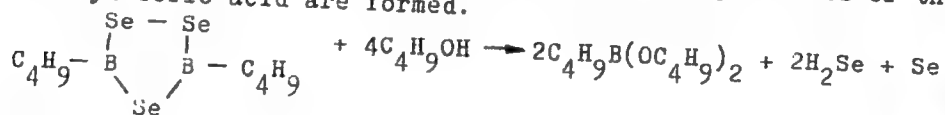
Effect of Selenium on Boron Trialkyls

SOV/62-59-2-30/40

action contains hydrogen and butylene. The reaction proceeds according to the following equation:



A similar reaction takes place on heating of triisobutyl boron with selenium, wherein 3,5-diisobutyl-3,5-diboron-1,2,4-triselenolane (II) is formed. 3,5-dialkyl-3,5-diboron-1,2,4-triselenolanes are yellow viscous liquids. They are very sensitive to atmospheric moisture and oxygen. By the influence of n-butyl alcohol upon 3,5-di-n-butyl-3,5-diboron-1,2,4-triselenolane metallic selenium, hydrogen selenide and n-butyl esters of the n-butyl boric acid are formed.



There are 3 references, 1 of which is Soviet.

Card 2/3

Effect of Selenium on Boron Trialkyls

SOV/62-59-2-30/40

ASSOCIATION: Institut organicheskoy khimii im. N. D. Zelinskogo Akademii  
nauk SSSR (Institute of Organic Chemistry imeni N. D. Zelinskiy  
of the Academy of Sciences, USSR)

SUBMITTED: July 9, 1958

Card 3/3



5(3)

AUTHORS: Mikhaylov, B. M., Shchegoleva, T. A. SOV/62-59-3-25/37

TITLE: Synthesis of Trialkyl Borons With Functional Substituents  
(Sintez bortrialkilov s funktsional'nyimi zamestitelyami)

PERIODICAL: Izvestiya Akademii nauk SSSR. Otdeleniye khimicheskikh nauk,  
1959, Nr 3, pp 546-547 (USSR)

ABSTRACT: This is a brief communication on the investigation of the reaction of diborane with vinyl-ethyl ether. Tri-(2-ethoxyethyl)-boron (I) was obtained in insufficient purity as a result of this reaction. Probably, in this case the reaction is complicated by side reactions. In reference 6 it was shown that the lowest alkyl group may be easily replaced by the highest one if the mixture of the corresponding trialkyl boron and olefin is heated and the easily volatile olefin is removed from the reaction mixture. The authors extended this reaction to unsaturated compounds with functional substituents. By heating triisobutyl boron with 2-chloro-1,1,2-trifluoroethyl allyl ether (II) tri-[3-(2'-chloro-1',1',2'-trifluoroethoxy)propyl] boron (III) was obtained in a yield of 52 %. Triisobutyl boron reacts also with unsaturated organosilicon compounds. By heating triisobutyl boron with allyl trimethyl silane or

Card 1/2

Synthesis of Trialkyl Borons With Functional  
Substituents

SOV/62-59-3-25/37

allyl trichlorosilane tri-(3-trimethyl silyl propyl) boron (IV) and tri-(3-chlorosilyl propyl)boron (V) were obtained accordingly in yields of 62-69 %. The investigations of the synthesis of functional trialkyl boron derivatives according to the mentioned method are continued. There are 7 references, 1 of which is Soviet.

ASSOCIATION: Institut organicheskoy khimii im. N. D. Zelinskogo Akademii nauk SSSR (Institute of Organic Chemistry imeni N. D. Zelinskiy of the Academy of Sciences, USSR)

SUBMITTED: July 9, 1958

Card 2/2

5(3)

AUTHORS:

Mikhaylov, B. M., Shchegoleva, T. A. SOV/62-59-8-8/42

TITLE:

Boron-organic Compounds. Communication 42: Alkylpyro-boro-acetic Anhydrides

PERIODICAL:

Izvestiya Akademii nauk SSSR. Otdeleniye khimicheskikh nauk, 1959, Nr 8, pp 1393-1396 (USSR)

ABSTRACT:

It was shown in the present paper that the mixed anhydrides of fatty acids and n-propylpyro boric acid normally formed under the influence of organic acids upon esters of n-propylchloro boric acid may be obtained much more easily by heating alkyl boric acid with the anhydride of acetic acid over a water bath. In this way it was possible to synthesize the anhydrides of n-propylpyro-boroacetic acid (I), isopropylpyro-boroacetic acid (II), and n-butylpyro-boroacetic acid (III) as well as isobutylpyro-boroacetic acid (IV), with yields ranging between 60 and 70% of the theoretical yields. The compounds obtained were investigated as to their different reactivities. It was shown that the anhydrides of alkylpyro-boroacetic acid easily react with the esters of alkyl boric acid at 160-185°, forming the anhydrides of alkyl boric acid. The anhydride of n-propylpyro boric acid forms a mixture of

Card 1/2

Boron-organic Compounds. Communication 42: Alkylpyro- SOV/62-59-8-8/42  
boroacetic Anhydrides

anhydrides of propyl and phenyl boric acids when heated with the esters of phenyl boric acid. The anhydride of n-propylpyro boric acid forms anhydride of propyl boric acid also with the esters of n-propylchloro boric acid. In the experimental part the reaction method is described. There is 1 Soviet reference.

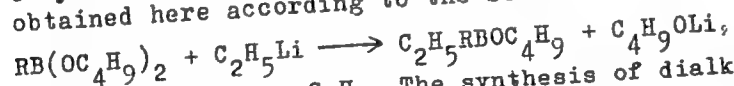
ASSOCIATION: Institut organicheskoy khimii im. N. D. Zelinskogo Akademii nauk SSSR (Institute of Organic Chemistry imeni N. D. Zelinskiy of the Academy of Sciences, USSR)

SUBMITTED: November 21, 1957

Card 2/2

5(3)  
 AUTHORS: Mikhaylov, B. M., Shchegoleva, T. A.  
 TITLE: Organoboron Compounds. XLVI. Dialkyl Boric Acids and Their Derivatives  
 PERIODICAL: Zhurnal obshchey khimii, 1959, Vol 29, Nr 9, pp 3130-3135 (USSR)

ABSTRACT: Alkylborates may serve for the synthesis of symmetrical and unsymmetrical dialkyl borates. The butyl esters of di-n-butyl- and n-propyl-n-butyl boric acid were synthesized by reaction of n-butyl lithium or n-propyl lithium with the n-butyl ester of n-butyl boric acid (Ref 11). Based on this method n-butyl-ethyl-n-butyl borate and n-butyl-ethyl-n-propyl borate were obtained here according to the scheme:



where  $R = C_4H_9$ , n.- $C_3H_7$ . The synthesis of dialkyl borates on the basis of alkyl borates can also be practiced with organo-magnesium compounds. Thus, a 45% yield of n-butyl-di-n.-propyl borate was obtained by reaction of n.-propyl magnesium bromide with n.-butyl-n.-propyl borate:

Card 1/3

SOV/79-29-9-68/76

Organoboron Compounds. XLVI. Dialkyl Boric Acids and Their Derivatives

$n\text{-C}_3\text{H}_7\text{B}(\text{OC}_4\text{H}_9)_2 + n\text{-C}_3\text{H}_7\text{MgBr} \rightarrow (n\text{-C}_3\text{H}_7)_2\text{BOC}_4\text{H}_9$ . The dialkyl boric acids resulted from saponification of dialkyl borates. The stability of dialkyl borates to hydrolyzing agents is conditioned by the nature of the arrangement of esters (for examples see references 8, 10). In this regard, n.-butyl dialkyl borates are much more stable than glycol esters (Refs 12, 13). The reaction of n.-butyl-di-n.-butyl borate with a 10% caustic soda solution with subsequent acidification is shown by scheme 1. Butyl alcohol is also completely eliminated together with water in the concentration of a salt solution (I) gently heated and distilled in vacuum. The crystalline residue is a sodium salt of di-n.-butylborenum acid (II) in analytically pure state. The reaction process of the formation of salt (II) is explained by scheme (2). Di-n.-butyl boric acid (Scheme 5) results from acidification of salt (II) after an intermediate stage of acid (V). Di-n.-propyl boric acid and n.-propyl-n.-butyl boric acid are colorless liquids tending to pass over into the corresponding anhydrides when they are heated. There are 14 references,

Card 2/3

SOV/79-29-9-68/76

Organoboron Compounds. XLVI. Dialkyl Boric Acids and Their Derivatives

4 of which are Soviet.

ASSOCIATION: Institut organicheskoy khimii Akademii nauk SSSR  
(Institute of Organic Chemistry of the Academy of Sciences,  
USSR)

SUBMITTED: July 10, 1958

Card 3/3

5.3700(B)

82103  
S/062/60/000/07/07/007  
B015/B054

AUTHORS: Mikhaylov, B. M., Shchegoleva, T. A., Blokhina, A. N.  
TITLE: Reaction of Tetra-n-butyl Mercapto Diborane <sup>1</sup> with  
Unsaturated Compounds  
PERIODICAL: Izvestiya Akademii nauk SSSR. Otdeleniye khimicheskikh nauk,  
1960, No. 7, pp. 1307-1309

TEXT: The olefin hydrocarbons (hexene-1, octene-1, styrene) react with tetra-n-butyl mercapto diborane at 70-80°C in the presence of pyridine under formation of the n-butyl esters of alkyl thioboric acids. The reaction between tetra-n-butyl mercapto diborane and propylene or isobutylene proceeds in a complicated way. On heating tetra-n-butyl mercapto diborane with propylene in the presence of pyridine in an autoclave at 70-80°C and 5-15 atm, the n-butyl ester of n-propyl thioboric acid as well as the n-butyl ester of di-n-propyl thioboric acid and tri-n-butyl thioborate are formed. Tetra-n-butyl mercapto diborane reacts with isobutylene in a similar way; a mixture consisting of the esters of isobutyl thioboric- and diisobutyl thioboric acid as well as tri-n-butyl

Card 1/2



Reaction of Tetra-n-butyl Mercapto Diborane With  
Unsaturated Compounds

82103  
S/062/60/000/07/07/007  
B015/B054

thioborate are formed. The formation of the esters of dialkyl thioboric acids and of thioborate is explained by the fact that tetra-n-butyl mercapto diborane symmetrizes to thioborate and di-n-butyl mercapto diborane, and the latter reacts with the olefins under formation of the corresponding esters of dialkyl thioboric acids. There are 3 Soviet references.

ASSOCIATION: Institut organicheskoy khimii im. N. D. Zelinskogo Akademii nauk SSSR  
(Institute of Organic Chemistry imeni N. D. Zelinskiy of the Academy of Sciences, USSR)

SUBMITTED: December 24, 1959

Card 2/2

11.5000  
5.3700

69509

AUTHORS:

Mikhaylov, B. M., Shchegoleva, T. A.

S/020/60/131/04/035/073

B011/B017

TITLE:

Synthesis and Some Transformations of Alkylmercaptodiboranes <sup>1</sup>

PERIODICAL:

Doklady Akademii nauk SSSR, 1960, Vol 131, Nr 4, pp 843-846 (USSR)

TEXT: The authors studied the reaction of diborane with n-propylmercaptan and n-butylmercaptan in ether solution at room temperature and with different ratio of the reagents. Tetraalkylmercaptodiborane is formed as a main reaction product if a mercaptan excess is present (see Scheme). These compounds show considerable resistivity: they do not change on longer storage, and may be distilled under vacuum. On distilling they turn into highly viscous liquids, but some hours later they become mobile again. The molecular weight of the dimeric form of dialkylmercaptoborane is by about 1.5 times higher than the cryoscopically determined molecular weight of tetraalkylmercaptodiboranes. Apparently, a partial dissociation of the produced alkylmercapto derivatives of diborane takes place in the benzene solution. In the reaction between diborane and n-butylmercaptan (ratio 1:2), much less tetra-n-butylmercaptodiborane is formed. Di-n-butylmercaptodiborane  $C_4H_9SBH_2BH_2SC_4H_9$  is formed as a main product. It is an unstable compound which is symmetrized on storing at room temperature into diborane and tetra-n-butylmercaptodiborane. The latter was identified on the basis of its capability of reacting with olefines at room temperature and of forming n-butyl ester of dialkyl-

Card 1/3

69509

Synthesis and Some Transformations of Alkylmercaptodiboranes

S/020/60/131/04/035/073  
B011/B017

thioboric acids (see Scheme). Furthermore, the yield in tri-n-propylboron, n-butyl ester of di-n-propylthioboric acid, and tetra-n-butylmercaptodiborane on passing propylene through the reaction mass of diborane with n-butylmercaptan is mentioned. The yield differed according to whether the mass was fresh, or stored for one night. The yield in di-n-butylmercaptodiborane is very low on the action of diborane on tetra-n-butylmercaptodiborane since equilibrium is established between the reagents and the final product (see Scheme). Polymers are known which were produced from diborane and methylmercaptan in the gas phase (Ref 2). The hydrogen atoms of tetraalkylmercaptodiboranes show much lower reactivity than diborane. Thus, the reaction of the former with mercaptan starts only at 50-60°, and proceeds energetically at the boiling temperature of mercaptan. In this connection, trialkylthioborate (see Scheme) is formed. On treating tetra-n-butylmercaptodiborane with water or alcohol at room temperature, no hydrogen is separated. Under the same conditions, N-trialkylborazols were obtained under the action of primary amines (ethylamine, n-butylamine) on tetra-n-butylmercaptodiborane. Apparently, a complex of dialkylmercaptoborane with amine (I) is formed during the first stage of the process. This complex is transformed into alkylmercaptoalkylaminoborane (II) with mercaptan being separated. An N-alkyl derivative of borazol is formed from (II) under precipitation of mercaptan. The high stability of the B-H bond becomes manifest in the reactions between tetraalkylmercaptodiboranes and olefines. The authors

Card 2/3

69509

Synthesis and Some Transformations of Alkylmercapto-  
diboranes

S/020/60/131/04/035/073  
B011/B017

succeeded in adding tetra-n-butylmercaptodiborane to olefine hydrocarbons by heating the reagents to 70° in the presence of pyridine. There are 6 references, 2 of which are Soviet. X

ASSOCIATION: Institut organicheskoy khimii im. N. D. Zelinskogo Akademii nauk  
SSSR  
(Institute of Organic Chemistry imeni N. D. Zelinskiy of the  
Academy of Sciences, USSR)

PRESENTED: December 22, 1959, by B. A. Kazanskiy, Academician

SUBMITTED: December 21, 1959

Card 3/3

MIKHAYLOV, B.M.; SHCHEGOLEVA, T.A.; SHASHKOVA, Ye.M.

Synthesis of alkylthioboric acid esters from trialkylborines and  
thioborates. Izv. AN SSSR. Otd. khim. nauk no. 5: 916-917 My '61.  
(MIRA 14:5)

1. Institut organicheskoy khimii im. N.D. Zelinskogo AN SSSR.  
(Boric acid) (Boron compounds)

SHCHEGOLEVA, T.A.; SHASHKOVA, Ye.M.; MIKHAYLOV, B.M.

Reactions of triethylthioborate with amines. Izv.AN SSSR.Otd.khim.  
nauk no.5:918-919 My '61. (MIRA 14:5)

1. Institut organicheskoy khimii im. N.D.Zelinskogo AN SSSR.  
(Boric acid) (Amines)

MIKHAYLOV, B.M.; SHCHEGOLEVA, T.A.

Synthesis of 1-n-butylmercaptoboracycloalkanes. Izv.AN SSSR, Otd.  
khim.nauk no.6:1142-1144, Je '61. (MIRA 14:6)

1. Institut organicheskoy khimii im. N.D.Zelinskogo AN SSSR.  
(Cycloalkanes)

25047  
S/062/61/000/006/009/010  
B118/3220

15.8150

AUTHORS: Mikhaylov, B. M., Shchegoleva, T. A., Shashkova, Ye. M.,  
Sheludyakov, V. D.

TITLE: Polymers and trimers of alkyl mercapto-boranes

PERIODICAL: Akademiya nauk SSSR. Izvestiya. Otdeleniye khimicheskikh  
nauk, no. 6, 1961, 1163

TEXT: The authors stated that the reaction of diborane with mercaptans (1:2) in ether results in polymer alkyl mercapto-boranes. The diborane reacts with methyl mercaptan, forming a solid polymer  $(CH_3SBH_2)_x$  which had been synthesized previously by A. Burg and R. Wagner (see below) without the use of a solvent. On reaction of ethyl mercaptan or n-butyl mercaptan with diborane, glass-like polymers of ethyl mercapto-borane  $(C_2H_5SBH_2)_x$  or of n-butyl mercapto-borane  $(n-C_4H_9SBH_2)_x$  are obtained after elimination of the ether by distillation. The polymers of ethyl mercapto-borane and n-butyl mercapto-borane are converted gradually at room temperature to the corresponding trimers of alkyl mercapto-borane. ✓

Card 1/3



25047

S/062/61/000/006/009/010

B118/B220

Polymers and trimers of alkyl...

The trimer of ethyl mercapto-borane ( $C_2H_5SBH_2$ )<sub>3</sub> has the following constants: boiling at 94-96°C (1 mm Hg);  $d_4^{20} = 0.9772$ ;  $n_D^{20} = 1.5323$ ; data obtained:  $H_{act}$  2.98; 2.90; B 14.37 %; 14.27 %; molecular weight (determined cryoscopically): 217.8; 220.2. The trimer of n-butyl mercapto-borane decomposes on vacuum distillation:  $d_4^{20} = 0.9376$ ;  $n_D^{20} = 1.5130$ ; data obtained:  $H_{act}$  2.17; 2.15; B 10.23; 10.32 %; molecular weight: 293.3; 294.9 corresponding to ( $C_4H_9SBH_2$ )<sub>3</sub>. The solid polymer of methyl mercapto-borane is stable; however, when it is dissolved in tetrahydrofuran, it is converted to the trimer of methyl mercapto-borane: boiling at 80-81°C (1.5 mm Hg);  $d_4^{20} = 1.0121$ ;  $n_D^{20} = 1.5483$ ; data obtained:  $H_{act}$  3.46; 3.37; B 17.80; 17.30 %; molecular weight: 182.5; 183.6 corresponding to ( $CH_3SBH_2$ )<sub>3</sub>. The trimers of alkyl mercapto-boranes are fairly stable against the action of air and water. There is 1 non-Soviet-

Card 2/3

Polymers and trimers of alkyl...

25047

S'062/61/000/006/009/010

B118/B220

bloc reference. The reference to the English-language publication reads as follows: A. Burg, R. Wagner, J. Amer. Chem. Soc. 76, 3307 (1954).

ASSOCIATION: Institut organicheskoy khimii im. N. D. Zelinskogo SSSR  
(Institute of Organic Chemistry imeni N. D. Zelinskiy USSR)

SUBMITTED: April 20, 1961

Card 3/3

89733

53700

1164 2209 1274

S/020/61/136/003/019/027  
B016/B052

AUTHORS: Shchegoleva, T. A. and Belyavskaya, Ye. M.

TITLE: Organoboron Compounds. Synthesis and Some Properties of  
Tri-(ethyl-mercapto)-diborane

PERIODICAL: Doklady Akademii nauk SSSR, 1961, Vol. 136, No. 3,  
pp. 638-641

TEXT: The authors report on the reaction of diborane and ethyl mercaptan in ethereal solutions at room temperature. They obtained a 60-70% yield of tri-(ethyl-mercapto)-diborane  $C_2H_5SBH_2BH(SC_2H_5)_2$  (I) independently of the quantitative proportion of reagents. (I) is a liquid which can be distilled in vacuum without decomposition, and does not dissociate in solutions. The substitution of the remaining three hydrogen atoms by ethyl mercapto groups is only of advantage between 110 and 150°C; in this case triethylthioborate  $B(SC_2H_5)_3$  (II) is formed in good yields. Ethyl amine complexes of di-(ethyl-mercapto)-borane  $(C_2H_5S)_2BH \cdot NH_2C_2H_5$  (III) and ethyl-mercapto-

Card 1/4

89733

Organoboron Compounds. Synthesis and Some  
Properties of Tri-(ethyl-mercapto)-diborane

S/020/61/136/003/019/027  
B016/B052

borane  $C_2H_5SBH_2 \cdot NH_2C_2H_5$  (IV) are formed first by the action of ethylamine on (I). Then ethyl-amino-ethyl-mercapto-borane (V) forms from (III) by separation of mercaptan, and is then condensed into N-triethyl borazole (VI) (see insert scheme). Ethyl mercaptan is also separated by (IV) which is converted into ethyl-amino-borane (VII). The latter is isolated from the reaction mixture especially in the form of a trimer (VIII). As regards its chemical properties, (VIII) is like the trimer of methyl-amino-borane (Ref. 2). However, there also exists a dimer (IX) of (VII) which together with (VIII) is contained in a liquid fraction with a boiling point of  $85-90^\circ/2$  mm Hg. When left standing, (IX) is converted into (VIII). (IX) differs from (VIII) as to its chemical properties: when heated, both are converted into N-triethyl-borazole (VI), in (IX), however, only at lower temperatures (partly already when distilled in vacuum), while in (VIII) no hydrogen is generated below  $140 - 150^\circ C$ ; at  $180^\circ C$  it is completely transformed. The behavior of (I) in propylene is like that of di(n-butyl-mercapto)-diborane in ethereal solutions (Ref. 1): the one half of its molecule which is more intensively hydrogenated accumulates on

Card 2/4

09155

Organoboron Compounds. Synthesis and Some  
Properties of Tri-(ethyl-mercapto)-diborane

S/020/61/136/003/019/027  
B016/B052

propylene already at room temperature. Thus di-n-propyl-thioboric acid ethyl ester  $(C_3H_7)_2BSC_2H_5$  forms. The other half of the molecule is dimerized into tetra-(ethyl-mercapto)-diborane  $(C_2H_5S)_2BHBH(SC_2H_5)_2$  (XI).

At room temperature (I) does not react with ethylene or octene. By boiling (I) in ethereal solutions with octane, the authors obtained an ester mixture of di-n-octyl-thioboric acid  $(C_8H_{17})_2BSC_2H_5$  (XII) and n-octyl-thioboric acid  $C_8H_{17}B(SC_2H_5)_2$  (XIII). This accumulation is accompanied by a symmetrization leading to the formation of (II) and tri-n-octylboron. There are 5 references: 2 Soviet and 2 US.

ASSOCIATION: Institut organicheskoy khimii im. N. D. Zelinskogo Akademii nauk SSSR (Institute of Organic Chemistry imeni N. D. Zelinskiy of the Academy of Sciences USSR)

PRESENTED: July 7, 1960, by B. A. Kazanskiy, Academician

SUBMITTED: July 4, 1960

Card 3/4

35570

S/062/62/000/003/006/C14  
B117/B144

11 1250  
5-2410

AUTHORS: Mikhaylov, B. M., Shchegoleva, T. A., and Bubnov, Yu. N.

TITLE: Organoboron compounds. Communication 92. Refractions of the bonds of boron with some elements

PERIODICAL: Akademiya nauk SSSR. Izvestiya. Otdeleniye khimicheskikh nauk, no. 3, 1962, 413-419

TEXT: Refractions of (B - C), (B - O), (B - N), (B - S), and (B - Cl) bonds were calculated from molecular refractions of various organoboron compounds having regard to hybridization according to Denbigh's method. Where possible, compounds of the type  $BX_3$  were used. Molecular refractions were determined from the Lorentz-Lorenz law. For the bonds B - C<sub>aliph.</sub> and B - O, the mean value of their refractions was found from molecular refractions of boron trialkyls and trialkyl borates:  $R_D = 1.93 \text{ cm}^3$  and  $R_D = 1.61 \text{ cm}^3$ , respectively. For B - C<sub>arom.</sub>, a mean value of  $R_D = 2.76 \text{ cm}^3$  was determined from the molecular refractions of aryl boric acids.

Card 1/3

Organoboron compounds...

S/062/62/000/003/006/014  
B117/5144

Molecular refractions of triamides and N-substituted alkyl-(diamino) borons produced a mean value of  $1.97 \text{ cm}^3$  for the refractions of B - N. The mean refraction value for the B - N bond in dialkyl-(amino) borons, their N-substituted and dialkyl boryl hydrazines was  $2.01 \text{ cm}^3$ . Thus, the mean refraction value of the B - N bonds may be assumed to be  $1.98 \text{ cm}^3$ . For the B - S bond in thioborates as well as in alkyl- and dialkyl thioboric esters, a mean refraction value of  $5.59 \text{ cm}^3$  was determined, which is somewhat higher than the value of  $5.20 \text{ cm}^3$  determined for this bond in dialkyl thioboric acids. Various organoboron chlorides were used for calculating the refractions of the B - Cl bond. As the production of these chlorides in pure form is difficult owing to their tendency towards disproportionation and their easy hydrolyzability, the values found showed high fluctuations and produced a mean value of  $R_D = 6.88 \text{ cm}^3$ . There are 13 tables and 27 references: 15 Soviet and 12 non-Soviet. The four most recent references to English-language publications read as follows:  
P. M. Christopher, T. J. Tully, J. Amer. Chem. Soc., 80, 6516 (1958);  
G. F. Hennion, P. A. McCusker, J. V. Marra, J. Amer. Chem. Soc., 80, 3481 (1958) and J. Amer. Chem. Soc. 81, 1768 (1959); D. Aubrey, M. Lappert,

Card 2/3

Organoboron compounds...

S/062/62/000/003/006/014  
B117/B144

J. Chem. Soc. 1959, 2927.

ASSOCIATION: Institut organicheskoy khimii im. N. D. Zelinskogo Akademii  
nauk SSSR (Institute of Organic Chemistry imeni  
N. D. Zelinskiy of the Academy of Sciences USSR)

SUBMITTED: October 16, 1961

Card 3/3



MIKHAYLOV, B.M.; SHCHEGOLEVA, T.A.; BUBNOV, Yu.N.

Organoboron compounds. Report No.92: Refractions of boron  
bonds with some elements. Izv.AN SSSR.Utd.khim.nauk no.3:  
413-419 Mr '62. (MIRA 15:3)

1. Institut organicheskoy khimii im. N.D.Zelinskogo AN SSSR.  
(Boron organic compounds) (Refractometry)

39573  
S/062/62/000/007/004/013  
B117/B180

5.541 2220  
AUTHORS: Mikhaylov, B. M., Shchegoleva, T. A., Shashkova, Ye. M., and  
Sheludyakov, V. D.

TITLE: Organoboron compounds. Report 102. Monoalkyl mercapto  
derivatives of borane

PERIODICAL: Akademiya nauk SSSR. Izvestiya. Otdeleniye khimicheskikh  
nauk, no. 7, 1962, 1218 - 1223

TEXT: The reactions of diborane and mercaptanes in ether solution were  
studied at room temperature. Independent of the component ratio, diborane  
and methyl mercaptane yielded a solid, storable polymer which dissolves in  
ether and benzene, and converts into a trimer in a solution of tetrahydro-  
furan. A viscous, colorless polymer was produced from diborane and ethyl  
mercaptane, independent of the component ratio. The reaction of diborane  
with n-propyl and n-butyl mercaptanes only yielded polymers at a ratio of  
1 : 2. Trimers of ethyl, n-propyl, and n-butyl mercapto boranes formed  
spontaneously from the corresponding polymers. The resulting trimers are  
a new type of organoboron compound. They are very stable, have a cyclic

Card 1/2

Organoboron compounds...

S/062/62/000/007/004/013  
B117/B180

structure, are virtually unaffected by air, not completely oxidized by hydrogen peroxide and are very slowly hydrolyzed by heating. They yield the corresponding borates by alcoholysis. This reaction is slow at room temperature, accelerating as the temperature rises. Alkyl mercaptoborane trimers and mercaptanes only react at 100 - 120°C, yielding large amounts of alkyl thioborates. 53% methyl thioborate and 89% ethyl thioborate were obtained by boiling a mixture of high-boiling mercaptane and trimer.

ASSOCIATION: Institut organicheskoy khimii im. N. D. Zelinskogo Akademii nauk SSSR (Institute of Organic Chemistry imeni N. D. Zelinskiy of the Academy of Sciences USSR)

SUBMITTED: January 30, 1962

Card 2/2

MIKHAYLOV, B.M.; SHELYDAYAKOV, V.D.; SHCHEGOLEVA, T.A.

Organoboron compounds. Report No.106: Reactions of alkyl mercapto-  
borane trimers with secondary and tertiary amines. Izv.AN SSSR.Otd.khim.  
nauk no.9:1559-1564 S '62. (MIRA 15:10)

1. Institut organicheskoy khimii im. N.D.Zelinskogo AN SSSR.  
(Boron organic compounds) (Amines)

MIKHAYLOV, B.M.; SHELU DAYAKOV, V.D.; SHCHEGOLEVA, T.A.

New types of boron salts. *Izv. AN SSSR. Otd. khim. nauk* no. 9: 1698-1699  
S '62. (MIRA 15:10)

1. Institut organicheskoy khimii im. N.D. Zelinskogo AN SSSR.  
(Boron--Compounds)

S/062/63/000/003/006/018  
B101/B186

AUTHORS: Shchegoleva, T. A., Shashkova, Ye. M., and Mikhaylov, B. M.

TITLE: Organoboron compounds. Communication 113. Reduction of alkyl thioborates to dialkyl mercapto-boranes

PERIODICAL: Akademiya nauk SSSR. Izvestiya. Otdeleniye khimicheskikh nauk, no. 3, 1963, 494 - 497

TEXT: It was found that ethyl thioborate, n-propyl thioborate, n-butyl thioborate, isopropyl thioborate, and tert-butyl thioborate react with  $\text{LiAlH}_4$  in nitrogen atmosphere at room temperature to give the corresponding dialkyl mercapto-boranes:  $4(\text{RS})_3\text{B} + \text{LiAlH}_4 \rightarrow 4(\text{RS})_2\text{BH} + \text{LiSR} + \text{Al}(\text{SR})_3$ . The yield is 75 - 85 %. Reaction of methyl thioborate with  $\text{LiAlH}_4$ , however, did not give dimethyl mercapto-borane but a stable complex. This was confirmed by the following reaction:  $(\text{CH}_3\text{S})_3\text{B} + \text{LiH} \rightarrow [(\text{CH}_3\text{S})_3\text{BH}]\text{Li}$ . The resultant lithium-trimethyl-mercapto-boronhydride is a colorless solid substance which is heat-resistant up to  $300^\circ\text{C}$  and decomposes to  $\text{LiCl}$ ,

Card 1/2

Organoboron compounds. ....

S/062/63/000/003/006/018  
B101/B186

methyl mercaptane and dimethyl mercapto-borane when equimolar quantities of HCl are added, Dimethyl mercapto-borane cannot be prepared in pure condition, as it is partially dimerized even by distillation in vacuo.

This dimerization: 
$$\begin{array}{c} \text{RS} \quad \text{SR} \quad \text{SR} \\ \diagdown \quad \diagup \quad \diagdown \\ \text{B} \quad \text{B} \\ \diagup \quad \diagdown \quad \diagup \\ \text{H} \quad \text{SR} \quad \text{H} \end{array}, \quad \bar{\nu} = 2470, 2416 \text{ cm}^{-1}, \text{ is } 42 \% \text{ for}$$
  
R = CH<sub>3</sub>, 17 % for R = i-C<sub>3</sub>H<sub>7</sub>, and 0 % for R = tert-C<sub>4</sub>H<sub>9</sub>.

ASSOCIATION: Institut organicheskoy khimii im. N. D. Zelinskogo Akademii nauk SSSR (Institute of Organic Chemistry imeni N. D. Zelinskiy of the Academy of Sciences USSR)

SUBMITTED: June 6, 1962

Card 2/2

S/062/63/000/003/007/018  
B101/B186

AUTHORS: Mikhaylov, B. M., Dorokhov, V. A., and Shchegoleva, T. A.

TITLE: Organoboron compounds. Communication 114. Reaction of dialkyl-mercapto-boranes with secondary amines

PERIODICAL: Akademiya nauk SSSR. Izvestiya. Otdeleniye khimicheskikh nauk, no. 3, 1963, 498 - 499

TEXT: The reaction of di-n-butyl mercapto-borane with diethyl amine in a molar ratio of 1:1 in nitrogen atmosphere at room temperature gave n-butyl mercapto-(diethyl-amino)-borane in 53 % yield. Under equal conditions n-butyl mercapto-(piperidino)-borane was obtained in 71 % yield by reaction of di-n-butyl mercapto-borane with piperidine. Di-n-butyl mercapto-borane and diisoamyl amine gave n-propyl mercapto-(diisoamyl-amino)-borane in 85 % yield. On reaction of two moles of secondary amine with one mole of dialkyl mercapto-borane, bis-(dialkyl-amino)-borane as formed. Di-n-propyl-mercapto-borane and diiso-amyl amine thus gave bis-(diisoamyl-amino)-borane in 80 % yield, and di-n-propyl mercapto-borane and diallyl amine gave bis-(diallyl-amino)-borane in 90 % yield.

Card 1/2



Organoboron compounds ....

S/062/63/000/003/007/018  
B101/B186

ASSOCIATION: Institut organicheskoy khimii im. N. D. Zelinskogo Akademii  
nauk SSSR (Institute of Organic Chemistry imeni N. D.  
Zelinskiy of the Academy of Sciences USSR)

SUBMITTED: June 6, 1962

Card 2/2

ACCESSION NR: AP3000122

S/0062/63/000/005/0816/0822

AUTHOR: Mikhaylov, B. M.; Shchegoleva, T. A.; Sheludyakov, V. D.

TITLE: Organic boron compounds. Report 117. Reactions of the amine complexes of alkylmercaptoboranes with halogen derivatives of hydrocarbons and HCl

SOURCE: AN SSSR. Izvestiya. Otdeleniye khimicheskikh nauk, no. 5, 1963, 816-822

TOPIC TAGS: reaction mechanisms, amine complexes of alkylmercaptoboranes, HCl, halogenated hydrocarbons

ABSTRACT: Di-(dimethylamine)borane salts were formed by the action of halogenated hydrocarbons and HCl on dimethylaminealkylmercaptoboranes. When complexes of alkylmercaptoboranes with tertiary amines reacted with alkyl halides, the alkylmercapto group was exchanged for a halide group, converting them into borane halide complexes. HCl cleaved the trialkylamine complexes of alkylmercaptoboranes, giving alkylmercaptoborane trimers and amine hydrochlorides. Dimethylamine-methylmercaptoborane reacted with dimethylamine hydrochloride to yield the chloride of di-(dimethylamine)borane. Orig. art. has: 11 equations.

ASSOCIATION: Institut organicheskoy khimii im. N. D. Zelinskogo, Akademii nauk SSSR (Institute of Organic Chemistry, Academy of Sciences SSSR)

~~Card 1/2~~

L 17098-63

Ps-4 RM/WW/JD/MAY

EPR/EWP(j)/EPF(c)/EWP(q)/EWT(m)/BDS AFFTC/ASD Pr-4/Pc-4/

S/062/63/000/004/008/022

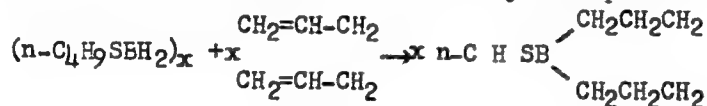
77  
76

AUTHOR: Mikhaylov, B. M., Shchegoleva, T. A., Sheludyakov, V. D., and  
Blokina, A. N.

TITLE: Organo-boron compounds. Report 116. Reactions of alkylmercapto-  
borane polymers with unsaturated compounds

PERIODICAL: Akademiy nauk SSSR. Izvestiya. Otdeleniye khimicheskikh nauk,  
no. 4, 1963, 646-651

TEXT: Inasmuch as various derivatives of diborane exhibit the capability  
to be added to unsaturated compounds the authors sought to study the behavior  
of alkylmercaptoborane polymers in relation to olefins and diene hydrocarbons.  
The addition of the n-butylmercaptoborane polymer to diallyl was performed,  
which results in the formation of 1-n-butylmercaptoboronocycloheptane:



Card 1/2

L 17098-63

S/062/63/000/004/C08/022

Organo-boron compounds.....

A polymer of methylmercaptoborane joins with olefins to form methy ethers of dialkylthioboric acid. In the action of isoprene on a polymer of methylmercaptoborane the product is 3-methyl-1-methylmercaptoborocyclopentane. A nitrogen atomosphere was used in all operations involving organo-boron compounds.

ASSOCIATION: Institut organicheskoy khimii im. N.D. Zelinskogo Akademii nauk SSSR (Institute of Organic Chemistry imeni N. D. Zelenskiy, Academy of Sciences USSR)

SUBMITTED: June 7, 1962

Card 2/2

SHCHEGOLEVA, T.A.; SHELUDYAKOV, V.D.; MIKHAYLOV, B.M.

Nature of the coordination compounds formed by boron and  
diborane halides in ether solutions. Dokl. AN SSSR 152 no.4:  
888-891 0 '63. (MIRA 16:11)

1. Institut organicheskoy khimii im. N.D. Zelinskogo AN SSSR.  
Predstavleno akademikom B.A. Kazanskim.

L 19490-65 EPF(c)/EPR/EWA(h)/EWP(j)/ENT(m)/T Pc-4/Pr-4/Ps-4/Peb RPL  
RM/WW/JW

ACCESSION NR: AP5002072

S/0062/64/000/002/0365/0367 31

AUTHOR: Shchegoleva, T. A.; Shashkova, Ye. M.; Kiselev, V. G.; Mikhaylov, B. M.<sup>B</sup>

TITLE: Hydroboration of dienes with chloroborane 7

SOURCE: AN SSSR. Izvestiya. Seriya khimicheskaya, no. 2, 1964, 365-367

TOPIC TAGS: organoboron compound, chloroborane, boron addition, diene boridation, diallyl, pentadiene, butadiene, borocyclopentane

ABSTRACT: In order to determine the effect of the nature of the diene on addition across the double bond, the authors studied the addition of chloroborane to diallyl, pentadien-1,4 and butadien-1,3<sup>12</sup> in ether solution at room temperature. Chromatography and degradation of the reaction products showed that diallyl adds primarily in the 1,6 position (74%), with smaller amounts of 1,5 and 2,5 addition products. Fractional distillation of this mixture resulted in good yields of pure 1-chloroborocyclopentane. Addition to pentadien-1,4 took place in both the 1,5 and 1,4 positions (53% and 47%, respectively), while addition to butadien-1,3 was mostly in the 1,4 position (75%), with 21% of the 1,3 addition product. The reaction conditions and yields are given. Orig. art. has: 2 chemical equations.

ASSOCIATION: Institut organicheskoy khimii im. N. D. Zelinskogo Akademii nauk  
Card 1/2

L 19490-65

ACCESSION NR: AP5002072

SSSR (Institute of Organic Chemistry, Academy of Sciences, SSSR)

SUBMITTED: 19Jul63

ENCL: 00

SUB CODE: OC

NO REF SOV: 001

OTHER: 003

Card 2/2

ACCESSION NR: AP4033385

S/0062/64/000/004/0632/0637

AUTHOR: Sheludyakov, V. D.; Shchegoleva, T. A.; Mikhaylov, B. M.

TITLE: Organic boron compounds.

Communication 129. Reaction of alkylmercaptoborane trimers with primary amines.

SOURCE: AN SSSR. Izvestiya. Seriya khimicheskaya, no. 4, 1964, 632-637

TOPIC TAGS: organic boron compound, alkylmercaptoborane trimer, alkylmercaptoborane trimer amine reaction, reaction mechanism, synthesis methylamine methylmercaptoborane complex, borane chloride

ABSTRACT: The reaction of alkylmercaptoborane trimers with primary amines was studied. Action of methylamine on methylmercaptoborane gives the methyl mercaptide of bis(methylamine)borane regardless of the reagent ration (trimer:amine of 1:6 or 1:3). The mechanism suggested is the formation of an intermediate neutral complex, methylamine-methylmercaptoborane, which reacts more rapidly with methylamine than the trimer. The less stable ethylmercaptide of bis(ethylamine)borane is formed similarly. These compounds exchange the mercaptide ion for the chloride ion under action of ether solutions of HCl:  $[H_2B(NH_2R')_2]SR + HCl \rightarrow [H_2B(NH_2R')_2]Cl +$

Card 1/2



ACCESSION NR: AP4033385

RSH. Similar exchange is effected with benzyl chloride. Normal-butylamine will not form the corresponding ethylmercaptide bis(n-butylamine)borone, only the complex  $C_2H_5SBH_2 \cdot NH_2C_4H_9$ . This will react with benzyl chloride to form the salt  $[H_2B(NH_2C_4H_9)_2]Cl$ . Borone salts with other amines in the capacity of addends were similarly formed by treating alkylmercaptoboranes with mixtures of the amine and benzyl chloride. The chlorides of bis(propyl, isopropyl, isobutyl, t-butyl, n-amyl, n-hexyl, or benzyl)borones are crystalline, stable, ether-insoluble materials. Orig. art. has: 2 tables and 12 equations.

ASSOCIATION: Institut organicheskoy khimii im. N. D. Zelinskogo Akademii nauk SSSR (Institute of Organic Chemistry, Academy of Sciences, SSSR)

SUBMITTED: 31Oct63

SUB CODE: OC

NO REF SOV: 002

ENCL: 00

OTHER: 001

Card 2/2

L 52601-65 EWT(m)/EPF(c)/ERR/ENP(j) Pc-4/Pr-4/Ps-4 RPL WW/RM

ACCESSION NR: AP5015854

UR/0062/64/000/012/2165/2170

AUTHOR: Mikhaylov, B. M.; Shchegoleva, T. A.; Sheludyakov, V. D.

TITLE: Organoboron compounds. Communication 132. Synthesis of cationic complexes from boron trihalides

SOURCE: AN SSSR. Izvestiya. Seriya khimicheskaya, no. 12, 1964, 2165-2170

TOPIC TAGS: organoboron compound, halogenated organic compound, organic synthetic process

Abstract: Under the action of dimethylamine on boron trichloride and boron tribromide in a 2:1 reagent ratio, dichloro-bis(dimethylamine)-boronium chloride and dibromo-bis(dimethylamine)boronium bromide are formed, respectively. The same salts are produced by the reaction of dimethylaminoboron dihalides with dimethylammonium salts. A boronium salt containing two different amines in the inner sphere, dichloro-dimethylaminepyridineboronium chloride, was synthesized by the action of pyridine hydrochloride on dimethylaminoboron dichloride, as well as

Card 1/2

L 52601-65

ACCESSION NR: AP5015854

by the reaction of boron trichloride, pyridine, and dimethylamine (in a 1:1 ratio). When ether and tetrahydrofuran solutions of boron trichloride were treated with ferric chloride or stannic chloride, dichlorobis(diethoxide)boronium and dichloro-bis(tetrahydrofuran)-boronium tetrachloroferrates and dichloro-bis(tetrahydrofuran)boronium hexachlorostannate were formed, indicating that in ether solutions boron trihalide exists not only in the form of etherates ( $BX_3 \cdot OR_2$ ), but also in the form of cationic complexes, in equilibrium with the etherates. Orig. art. has 7 formulas.

ASSOCIATION: Institut organicheskoy khimii im. N. D. Zelinskogo Akademii nauk SSSR  
(Institute of Organic Chemistry, Academy of Sciences, SSSR)

SUBMITTED: 16Feb63

ENCL: 00

SUB CODE: OC, GC

NO REF SOV: 007

OTHER: 011

JPRS

Card 2/2

SHCHESNIEV, T.S., SHELEBYAKOV, V.G.; MIKHAYLOVA, B.M.

Organoboron compounds. Part 151: Kinetics of the hydrolysis of  
boron cation complexes. Zhur. ob. khim. 35 no.6:1066-1073  
Ja '65. (MIRA 18:6)

SHCHEGOLEVA, T.A., SHASHKOVA, Ye.N.; KISELEV, V.G.; MIKHAYLOV, B.M.

Organoboron compounds. Part 158: Hydroboration of dienes by  
n-butylmercaptoborane. Zhur. ob. khim. 35 no.6:1078-1083  
Je '65. (MIRA 18:6)

1. The first of the two main points of the report is that the  
Soviet Union is a major threat to the United States.

2. The second point is that the Soviet Union is a major threat to the United States.

GLAZOVA, O. I., doktor med. nauk; IZRAELIT, S. S.; SHCHEGOLEVA, T. G.;  
LEIN, B. N.

Diagnosis of the active phase of the cardiac form of rheumatic  
fever. Terap. arkh. no.12:30-35 '61. (MIRA 15:2)

1. Iz terapevticheskoy kliniki (zav. - prof. P. I. Sukhinin) i  
laboratorii (zav. - kandidat meditsinskikh nauk V. V. Novosel'-  
skaya) Moskovskogo nauchno-issledovatel'skogo instituta skoroy  
pomoshchi imeni Sklifosovskogo.

(RHEUMATIC HEART DISEASE)

AUTHORS: Brynov, I. M. and Shchegoleva, T. V. 126-5-3-31/31  
TITLE: Nature of the Etching Patterns in an Ageing Alloy Al-Zn-Cu  
(Priroda figur travleniya v stareyushchem splave Al-Zn-Cu)  
PERIODICAL: Fizika Metallov i Metallovedeniye, 1957, Vol 5, Nr 3,  
pp 586-587 (USSR)  
ABSTRACT: According to Hirsch and Forte (Refs. 1 and 2) the etching  
patterns in crystals of various substances are associated  
with entry to the surface of spiral and boundary  
dislocations. During electron microscopic investigations  
of the alloy Al-Zn-Cu, after preliminary rolling and  
homogenization annealing, spiral etching patterns of cubic  
shape were detected by the authors on the etched surface  
(Figs. 1 and 2, p. 587). After partial ageing of the  
alloy (which did not result in intensive hardening) clear  
bright spots could be seen at the steps of the etching  
patterns (Fig. 2) which corresponded either to the zones  
of Guinier-Preston or to the particles of the separating  
out phase. The observed spirals could not be associated  
with dislocations having a single Burgers vector. On the  
basis of the features of the technique of oxide imprints  
which was applied in the given case, it can be assumed  
Card 1/3 that the etching steps can be detected by means of an



Nature of the Etching Patterns in an Ageing Alloy Al-Zn-Cu 126-5-5-51/31

electron microscope only for the spiral dislocations for a Burgers vector of at least 15-20 Å. In reality the Burgers vector in the given case amounts to several hundreds of Angstrom. This follows from the analysis of spectroscopic exposures as well as from the fact that the steps are clearly visible from the oxide imprints. It can, therefore, be concluded that each etching pattern is linked with protruding to the surface of the alloy of several larger or gigantic spiral dislocations which are parallel to the cubic axes of the crystal. (In view of the fact that Bontinek, W. (Ref.5) detected helinoidal dislocations in  $\text{CaF}_2$ , the possibility arose to associate spiral etching patterns with helinoidal and prismatic dislocations). Usually in the centre of each phase of the etching pattern not one but several (mostly three) spiral dislocations of a single sign will occur. However, the complexity of some of the spiral etching patterns leads to the assumption that in a number of cases dislocations of opposite signs take place at the face centres. Thus, contrary to existing theoretical conceptions (Ref.2) on the effect that it is not justified

Card 2/3

Nature of the Etching Patterns in an Ageing Alloy Al-Zn-Cu 126-5-3-31/31

to assume the presence of major dislocations in metals, the authors of this paper detected gigantic dislocations. Such dislocations, with Burgers vectors of several hundred Angstroms, were detected earlier in metals only by Amelinx (Ref.3) and Steinberg (Ref.4). Amelinx observed it on gold crystals grown by depositing gold from a solution of germinations of NaCl; Steinberg observed it on titanium crystals produced electrolytically. In both cases the appearance of gigantic spiral dislocations can be caused by the pertaining specific conditions, for instance growth on foreign body crystals. Amelinx pointed out that in his experiments, gold crystals could either follow the spiral dislocations of the common salt or appear due to major non-correspondence of the crystal lattices of gold and NaCl. In the here described experiments such conditions have apparently been made impossible.

Card 3/3 There are 2 figures and 5 references, 1 of which is Soviet, 4 English.

Note: This is a complete translation.

ASSOCIATION: Institut fiziki metallov Ural'skogo filiala AN SSSR  
(Institute of Metal Physics, Ural Branch of the Ac.Sc.USSR)

SUBMITTED: April 11, 1957

1. Aluminum-copper-zinc alloys--Aging 2. Aluminum-copper-zinc alloys  
--Crystal structure 3. Crystals--Physical properties

126-5-3-31/31

66227

SOV/126-8-3-10/33

18.1210  
AUTHORS:

Buynov, N.N., Shchegoleva, T.V., Rakin, V.G.,  
Komarova, M.F. and Zakharova, R.R.

TITLE:

Electron Microscopic Investigation of Etch Figures in  
Age Hardening Aluminium Alloys

PERIODICAL:

Fizika metallov i metallovedeniye, 1959, Vol 8, Nr 3,  
pp 387-393 (USSR)

ABSTRACT:

The results of an electron microscopic investigation of dimensions, form and structure of etch figures in age hardening aluminium alloys are discussed. In the table on p 388, data of the dimensions and shape of the etch figures for various alloys are given. The dimensions of the figures change within very wide limits from several microns to a few tenths. It is characteristic that for the majority of quenched, slightly aged specimens the etch figures are straight-sided (Fig 1) and for the hardened alloys they have an oval shape (Fig 2). Their dimensions decrease in relation to time and artificial ageing, when the hardness of the alloys increases. In Fig 3, an electron micrograph of an Al-Zn-Cu (10% Zn and 0.5% Cu) alloy, deformed by compression by 15% and aged at 180°C for 6 hours, is shown

Card 1/2

66227

Electron Microscopic Investigation of Etch Figures in Age Hardening  
Aluminium Alloys

SOV/126-8-3-10/33


Spiral steps can be seen. Fig 4 is an electron micrograph of an Al-Cu (4% Cu) alloy aged at 220°C for 5 min. Craters can be seen at the top of octahedra, suggesting screw dislocations. Fig 5 shows scheme for the layout of primary mosaic blocks in the crystalline alloy; the possible axes along which new blocks can form are shown by arrows. The authors arrive at the following conclusions: (1) The shape and dimensions of etch figures in aluminium alloys change with the time and temperature of ageing. (2) The relationship between etch figures and large screw or spiral dislocations justifies the assumption that they correspond to mosaic blocks. There are 5 figures, 1 table and 17 references, 7 of which are Soviet, 1 German, 1 Dutch and 8 English.

ASSOCIATION: Institut fiziki metallov AN SSSR (Institute of Metal Physics AS USSR)

SUBMITTED: August 12, 1958

Card 2/2

AUTHORS: Buynov, N.N. and Shchegoleva, T.V. SOV/126-8-3-21/33  
TITLE: A Few Characteristics of the Distribution of Etch  
Figures in an Al-Zn-Cu Alloy  
PERIODICAL: Fizika metallov i metallovedeniye, 1959, Vol 8, Nr 3,  
pp 455-457 (USSR)  
ABSTRACT: On studying the electrolytically polished and chemically  
etched (aqua regia) surface of an Al-Zn-Cu alloy  
(10% Zn, 0.5% Cu) which had been deformed in  
compression by less than 1%, spirals were observed  
which may possibly illustrate either Frank and Read's  
mechanism (Ref 1) or the existence of screw or helical  
dislocations. Beside them loops were observed, which  
were similar in shape to dislocation loops emitted by  
Frank and Read sources. They were observed not only  
within the grains (Fig 1 and 2) but also in grain  
boundaries (Fig 3). In the first case they are met with  
more frequently in groups, each of which contain from  
2 to 7 loops. In those cases where the loops are not  
continuous, they have the shape of hooks, the ends of  
which are bent inwards (Fig 1 to 6), which points to  
their non-accidental origin. Light photographs in a



Card 1/2

A Few Characteristics of the Distribution of Etch Figures in an  
Al-Zn-Cu Alloy

SOV/126-8-3-21/33

dark background (Fig 3) show that the loops consist of etched figures. However, there is one characteristic in the distribution of the loops which makes it doubtful as to whether they were formed due to the work of Frank and Read sources. In the central portion of several loop groups, a few small hooks with different orientation are observed in each group (in Fig 1 shown by an arrow). This peculiarity is difficult to understand if one takes Frank and Read's dislocation propagation as a basis and the authors of this paper have come to the conclusion that these loops are due to local stresses in various portions of the alloy. Gratitude is expressed to A.N.Orlov for discussion of the results of this paper. There are 6 figures and 4 references, 2 of which are Soviet and 2 English.

ASSOCIATION: Institut fiziki metallov AN SSR (Institute of Metal Physics AS USSR)

SUBMITTED: August 12, 1958

Card 2/2

69696

S/126/60/009/03/019/033  
E091/E435

18.1285

AUTHORS: Lerinman, R.M., Shchegoleva, T.V., Kushakevich, S.A.  
and Selitskaya, S.I.

TITLE: Electron Microscopic Investigation of Structural  
Transformations in Titanium-Manganese and Titanium-  
Chromium Alloys

PERIODICAL: Fizika metallov i metallovedeniye, 1960, Vol 9, Nr 3,  
pp 437-440 (USSR)

ABSTRACT: The transformation of the  $\beta$ -phase on tempering quenched Ti-Mn and Ti-Cr alloys were studied. The following binary alloys, containing elements which stabilize the  $\beta$ -phase, were used for the investigation: Ti-Mn (10.5% Mn) and Ti-Cr (9.4% Cr). The alloys were prepared from titanium sponge of TGO quality, manganese of MR1 and chromium of KhO quality. Ingots were prepared by double vacuum melting. For the alloy containing Mn, the second fusion was carried out in argon. The composition of the alloys is shown in the table on p 438. The ingots were deformed by hot rolling and forging and the alloys were water quenched from 850°C (ie from the  $\beta$ -region). The time of heating prior to quenching was 30 minutes. Tempering was carried

Card 1/3

69696  
S/126/60/009/03/019/033  
E091/E435

Electron Microscopic Investigation of Structural Transformations  
in Titanium-Manganese and Titanium-Chromium Alloys

out by soaking for 1 to 25 hours at 400 to 550°C and cooling in air. In order to reproduce the structures of the alloys, single-stepped angular prints (replicas) were prepared (Ref 10). The specimens were first chemically polished in anhydrous boiling ortho-phosphoric acid for 1 to 2 minutes. They were then etched in a mixture of 20% HF, 20% HNO<sub>3</sub> and 60% glycerin. The etching time varied from a few seconds to one minute. Apart from the electron microscopic investigation, hardness tests were made on a Rockwell machine with a diamond indenter, using a load of 150 kg. In Fig 1a, 1b and 1c, the microstructures of specimens of Ti-10.5% Mn alloys as tempered at 400°C for 1, 5 and 25 hours, respectively, are shown; in Fig 1g, 1d and 1e, those of similar specimens tempered at 550°C for 1, 10 and 25 hours, respectively. Fig 2 shows the microstructure of a Ti-9.4% Cr alloy (a - after quenching and tempering at 400°C for one hour; b - after quenching and tempering at 500°C for 25 hours). From the above microstructures

Card 2/3



69696  
S/126/60/009/03/019/033  
E091/E435

Electron Microscopic Investigation of Structural Transformations  
in Titanium-Manganese and Titanium-Chromium Alloys

it can be seen that an  $\omega$ -phase appears in Ti-Cr and Ti-Mn alloys after quenching and tempering at 400°C. It has the shape of very finely dispersed platelets, 300-400 Å thick. Periodically, chains of equiaxed particles and individual equiaxed particles can be observed which point to the fact that the  $\omega$ -phase has an equiaxed shape from the very moment of its formation. Gratitude is expressed to Yu.A. Bagaryatskiy and V.I. Dobatkin for the discussion of the results of this work. There are 2 figures, 1 table and 10 references, 7 of which are English, 2 French and 1 Soviet.

ASSOCIATION: Institut fiziki metallov AN SSSR  
(Institute of Physics of Metals AS USSR)

SUBMITTED: April 22, 1959

Card 3/3

4

MIKHAYLOV, D.M., FIDOTOV, N.S., SHCHEGOLEVA, T.A., SHELUDYAKOV, V.D.

Cation complexes of boron. Dokl. AN SSSR 145 no. 2:320-323 71  
163. (MIRA 15:17)

1. Institut organicheskoy khimii imeni N.D. Zelinskogo AN SSSR.  
Predstavleno akademikom B.A. Kazanskim.  
(Boron compounds) (Metal ions)

LERINMAN, R.M., SHCHEGOLEVA, T.V.; PAVLOVA, G.V.; ADOLINA, T.I.

Electron microscopy of plastic deformations in aluminum-silver alloys. Fiz. met. i metalloved. 13 no.4:623-630 O '64. (MIRA 13:4)

1. Institut fiziki metallov AN SSSR.

GRANDBERG, I.I.; DIN VEY-PY; SHCHEGOLEVA, V.I.; KOST, A.N.

Pyrazoles. Part 18: Dehydrogenation of 3-hydroxy- and 3-amino-pyrazolines with sulfur. Zhur.ob.khim. 31 no.6:1892-1896 Je '61.  
(MIRA 14:6)

1. Moskovskiy gosudarstvennyy universitet.  
(Pyrazoline) (Dehydrogenation)